

Exceeding Expectations

January 7, 2014

Kale Horton/Josephine Newton-Lund

USACE, Kansas City District

ATTN: CENWK-PM-E, Room 463

601 E. 12th, Federal Building Kansas City, MO 64106-2896

RE: Draft Final Site-Specific Work Plan

Former St. Louis Ordnance Plant

Regional Long Term Operations/Long Term Monitoring U.S. Army Corps of Engineers, Kansas City District

Contract No. W912DO-13-D-3000, Task Order 0004

USACE Project Manager: Kale Horton

Dear Mr. Horton/Ms. Newton-Lund:

HydroGeoLogic, Inc. (HGL) is pleased to submit one hard copy and one electronic copy of the Draft Final Site-Specific Work Plan for the Former St. Louis Ordnance Plant, Regional Long Term Operations/Long Term Monitoring. This document was prepared in accordance with the Task Order 0004 Performance Work Statement, HGL's Proposal dated May 17, 2013, and comments received on the Draft Site-Specific Work Plan. Hard copies and electronic copies have also been sent to the personnel copied on this letter.

Should you have any questions or comments, please contact me at 913-647-2536.

Sincerely,

Chris Williams, P.G.

HGL Task Order Manager

DTYY

40467550 Superfund 3,0

Enclosures

DUDI

Barry McFarland, 88th RSC – one electronic copy as requested Jonathan Harrington, AEC – one electronic copy (was unable to obtain his address) Jim Harris, MDNR – one hard copy and one electronic copy Matt Jefferson, EPA Region 7 - one hard copy and one electronic copy

DRAFT FINAL SITE-SPECIFIC WORK PLAN THE FORMER ST. LOUIS ORDNANCE PLANT ST. LOUIS, MISSOURI

REGIONAL LTO/LTM FOR SEVEN INSTALLATIONS

Prepared for:



U.S. Army Corps of Engineers Kansas City District

Contract No. W912DQ-13-D-3000 Task Order 0004

Prepared by:

HydroGeoLogic, Inc. 6340 Glenwood Street, Suite 200 Building #7 Overland Park, KS 66202

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LIST OF ACRONYMS AND ABBREVIATIONS

APP Accident Prevention Plan

CENWK U.S. Army Corps of Engineers, Kansas City District

CM Construction Manager

CQCS Contractor Quality Control Supervisor

FSP Field Sampling Plan

HGL HydroGeoLogic, Inc.

IDW investigation-derived waste

LTM long term monitoring LTO long term operations

OU operable unit

PDB passive diffusion bag PM Project Manager

PMP Project Management Plan
PPE personal protective equipment
PWS performance work statement

QAPP Quality Assurance Project Plan

QC quality control QCP Quality Control Plan

SLOP St. Louis Ordnance Plant SSHO Site Safety and Health Officer SSHP Site Safety and Health Plan

TL Technical Lead TO task order

10 task order

USACE U.S. Army Corps of Engineers

VOC volatile organic compound

DRAFT FINAL SITE-SPECIFIC WORK PLAN THE FORMER ST. LOUIS ORDNANCE PLANT ST. LOUIS, MISSOURI

REGIONAL LTO/LTM FOR SEVEN INSTALLATIONS

1.0 INTRODUCTION

This Site-Specific Work Plan describes Long Term Operation (LTO)/Long Term Monitoring (LTM) activities to be implemented at Operable Unit (OU)-1 at the former St. Louis Ordnance Plant (SLOP), in St. Louis, Missouri. This work is being conducted by HydroGeoLogic, Inc. (HGL) under U.S. Army Corps of Engineers (USACE), Kansas City District (CENWK) contract number W912DQ-13-D-3000, task order (TO) 0004, Regional LTO/LTM. The former SLOP is one of seven installations covered by the TO. The former SLOP is located at 6400 Stratford Avenue on the western boundary of the city limits of St. Louis, Missouri (Figure 1.1).

This Work Plan consists of the following component plans:

- Field Sampling Plan (FSP) provided in TAB 1;
- Quality Assurance Project Plan (QAPP) provided in TAB 2; and
- Site Safety and Health Plan (SSHP) Addendum specific to the former SLOP provided in TAB 3.

The FSP and QAPP detail the sampling and operational activities (if applicable) and the associated analytical requirements. These documents include appendices and attachments that provide such information as field forms and laboratory documentation. Copies will be provided to the analytical laboratory and appropriate field personnel, and may be used as an audit guide for field and laboratory work. The SSHP Addendum describes the specific safety and health measures to be implemented at the former SLOP during the field activities, and references the Regional Accident Prevention Plan (APP)/SSHP for TO 0004 (HGL, 2013c).

The other regional TO 0004 documents, which are incorporated by reference, are:

- Project Management Plan (PMP) (HGL, 2013a), and
- Quality Control Plan (QCP) (HGL, 2013b).

2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

The following sections describe the organizational structure that will be implemented to ensure that tasks and activities meet the project objectives and the specific requirements outlined in the Performance Work Statement (PWS). A detailed discussion of the project team, their contact information, the organizational chart, and resumes are provided in the PMP. Brief descriptions of the roles and responsibilities of key personnel are listed in this section.

2.1 PROJECT MANAGER

The duties and responsibilities of the Project Manager (PM) include the following:

- Overseeing contract execution;
- Documenting overall conformance to project requirements and specifications, including technical, cost, and schedule;
- Designating the Technical Lead (TL) and technicians;
- Reviewing required submittals; and
- Allocating sufficient resources to ensure successful completion of the tasks in the PWS.

2.2 TECHNICAL LEADS

TLs will report directly to the PM. The duties and responsibilities of the TLs include the following:

- Initiating project planning and implementation of project activities at the TO level;
- Managing the budget and schedule for their assigned project component, and documenting that contract requirements are satisfied, with concurrence from the PM;
- Managing field activities, including direction of project staff and subcontractors in accordance with requirements of the contract documents;
- Tracking proposed changes to the performance objectives for the overall project;
- Communicating directly with the PM regarding project execution and accountability;
- Coordinating with the Contractor Quality Control Supervisor (CQCS) to document compliance with standard protocols and procedures as well as implementation of the project plans;
- Coordinating with the Site Safety and Health Officer (SSHO) to implement the APP/SSHP and site-specific SSHP addendum; and
- Procuring equipment, material, and supplies.

2.3 CONSTRUCTION MANAGER

The Construction Manager (CM) will report directly to the PM. The duties and responsibilities of the CM include the following:

- Supports the PM and TL during construction activities, and communicates with them directly regarding project execution and accountability.
- Monitors construction work progress and schedule, and advises PM and TLs of variances.
- Directs all site construction activities to document conformance to the approved work plans.
- Coordinates with the CQCS to document compliance with standard protocols and procedures, and federal, state and local laws and regulations.
- Supervises on-site subcontractors, inspects field equipment, and evaluate/accepts ongoing fieldwork.
- Maintains a record of site personnel and work completed.
- Evaluates and troubleshoots construction activities, and identifies opportunities for optimization.

2.4 CORPORATE QUALITY ASSURANCE MANAGER

The Corporate Quality Assurance Manager has overall responsibility and authority for development and management HGL's Quality Control (QC) Program.

2.5 CONTRACTOR QUALITY CONTROL SUPERVISOR

The duties of CQCS include the following:

- Supervising the QC aspects of the project to document compliance with contract plans and specifications as defined in the QCP;
- Managing the QCP;
- Maintaining communication between project management and project team members;
- Approving submittals and supervising QC procedures; and
- Acting as the primary spokesman on quality matters when interfacing with external organizations.

2.6 SAFETY AND HEALTH MANAGER

The Safety Health Manager will be responsible for the following items:

- Recommending changes to engineering controls, work practices, and personal protective equipment (PPE);
- Coordinating with the SSHO for any on-site training needs;
- Consulting with the SSHO for on-site emergencies;
- Providing on-site consultation as needed to guide implementation of the SSHP and sitespecific addendum;

- Coordinating any modifications to the Regional SSHP and Site-Specific Addendum with the PM, SSHO, and the Contracting Officer or Contracting Officer's Representative; and
- Providing continued support for upgrading or downgrading the level of PPE.

2.7 SITE SAFETY AND HEALTH OFFICER

Specific job-related Activity Hazard Analyses have been generated for each major field activity and are provided in Appendix B of the regional APP/SSHP (HGL, 2013c). The role and responsibilities of the SSHO are detailed in the APP/SSHP and summarized below:

- Implementing and enforcing the APP/SSHP, and site-specific SSHP addendum (provided at TAB 3);
- Holding daily tailgate safety meetings during periods when fieldwork is under way;
- Documenting compliance with federal, state, and Occupational Safety and Health Administration safety and health regulations;
- Coordinating modifications to the safety and health plans with the Safety and Health Manager, TLs, and the PM; and
- Maintaining the project Health and Safety Records and Logbook.

3.0 SUMMARY OF WORK

3.1 LTO/LTM ACTIVITIES

The former SLOP operated from 1941 to 1945 as a small arms ammunition production facility, producing primarily .30- and .50-caliber ammunition. Plant Area No. 2 (located west of Goodfellow Boulevard) encompassed 27.68 acres at the former SLOP. The former Hanley Area consists of the 14.68 acres at the northeastern end of Plant Area No. 2 at the intersection of Stratford Avenue and Goodfellow Boulevard. Production at Plant Area No. 2 consisted of blending primary explosives, incendiary compounds, and tracer charging .30- and .50-caliber projectiles as part of the assembly of the final product (Conti and CH2MHill, 2012). Figure 1.1 shows the site layout within the former Hanley Area.

The current regulatory status of the Site is LTO/LTM to monitor groundwater and maintain the remedy components. The definable features of work for the LTO/LTM at the former SLOP are as follows:

- Conduct Project Management activities;
- Conduct quarterly sampling of 12 monitoring wells through 2014 and annual sampling from 2015 through 2017;
- Inspect and maintain the monitoring wells;
- Manage disposal of investigation-derived waste (IDW) purge water;
- Maintain the former SLOP data in the regional LTO/LTM project database; and
- Prepare quarterly and annual reports during the 5-year period of performance.

The groundwater sampling consists of employing passive diffusion bag (PDB) sampling techniques at all 12 wells. Based on Section 5.3 in the Final Long-Term Management/Land Use Control Implementation Plan, the wells are sampled for the following volatile organic compounds (VOCs) (Conti and CH2MHill, 2012):

- Benzene
- Naphthalene
- Carbon tetrachloride
- Chloroform
- 1,1,1,2-tetrachloroethane
- 1,1,2,2-tetrachloroethane
- 1,2-dichloroethane
- 1,1,2-trichloroethane
- cis-1,2-dichloroethene
- trans-1,2-dichloroethene
- Tetrachloroethene
- Trichloroethene
- Methylene chloride

• Vinyl chloride

Additionally, the wells will be inspected during the sampling events for conditions such as broken pads, damaged well covers, and missing locks. These maintenance issues will be addressed as they occur.

PDBs will be used; therefore, purge water will not be generated from sampling activities. However, a minimal amount of sample material may remain in the PDB after the sample bottles have been filled. Any remaining water will be containerized. Aqueous waste will be transferred into a U.S. Department of Transportation–approved 55-gallon drum, characterized, and discharged into the on-site combined sanitary and stormwater sewer (following Metropolitan Sewer District approval).

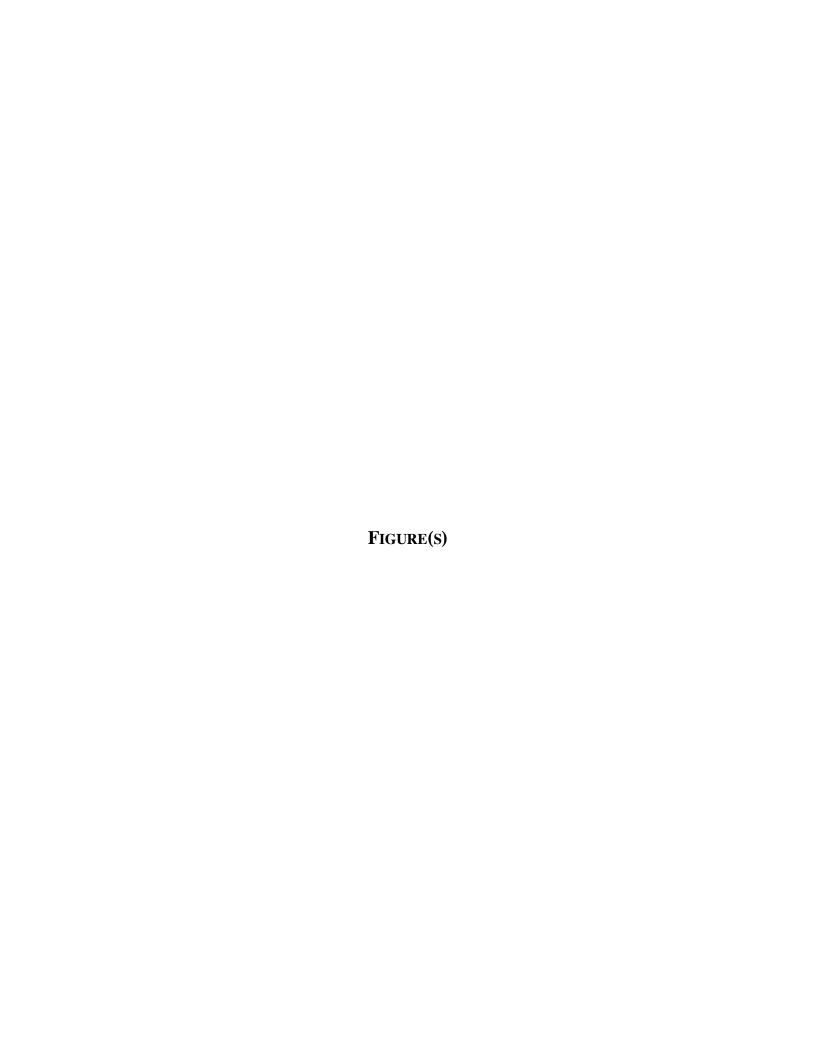
3.2 OPTIMIZATION APPROACH

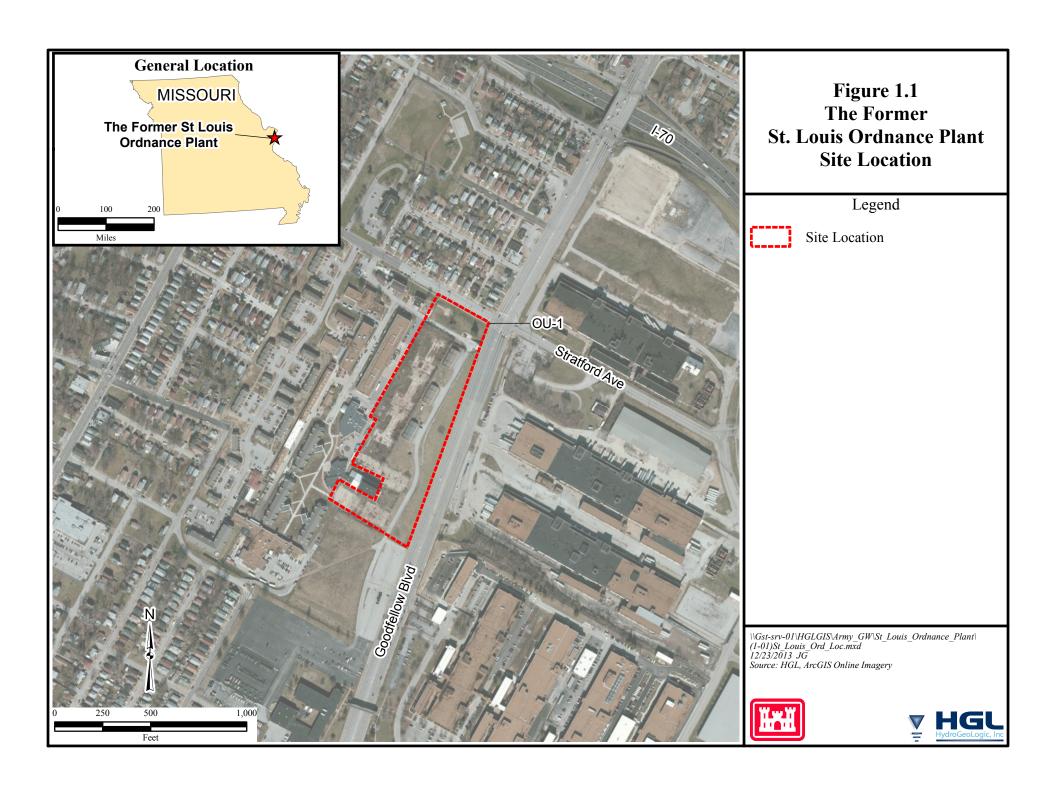
An LTM optimization evaluation will be conducted at the former SLOP after eight sampling events have been conducted. If three consecutive rounds of groundwater monitoring indicate that concentrations of site contaminants of concern fall below remediation goals and risk-based thresholds in monitoring wells within the monitoring network, then project stakeholders will consider terminating the groundwater monitoring program (Conti and CH2MHill, 2012).

Four of the eight sampling events needed to perform optimization have been completed. Optimization also may lead to fewer sampling events per year, or a reduced number of wells for LTM. HGL will present the results of the optimization evaluation after the eighth quarterly sampling event to be completed in July 2014.

4.0 REFERENCES

- Conti and CH2MHill, 2012. Final Long-Term Management/Land Use Control Implementation Plan Operable Unit 1, St. Louis Ordnance Plant Former Hanley Area, St. Louis, Missouri. September.
- HydroGeoLogic, Inc. (HGL), 2013a. Draft Project Management Plan, Regional Long Term Operations/Long Term Monitoring. September.
- HGL, 2013b. Draft Quality Control Plan, *Regional Long Term Operations/Long Term Monitoring*. September.
- HGL, 2013c. Draft Accident Prevention Plan/Site Safety and Health Plan. Regional Long Term Operations/Long Term Monitoring. September.





DRAFT FINAL FIELD SAMPLING PLAN THE FORMER ST. LOUIS ORDNANCE PLANT ST. LOUIS, MISSOURI

REGIONAL LTO/LTM FOR SEVEN INSTALLATIONS

Prepared for:



U.S. Army Corps of Engineers Kansas City District

Contract No. W912DQ-13-D-3000 Task Order 0004

Prepared by:

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January 2014

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LIST OF ACRONYMS AND ABBREVIATIONS

CENWK U.S. Army Corps of Engineers, Kansas City District

CoC chain of custody

contaminant of concern COC

DFW definable feature of work

deionized water DI

U.S. Environmental Protection Agency **EPA**

FSP Field Sampling Plan

HGL HydroGeoLogic, Inc.

IDW investigation-derived waste

Interstate Technology Regulatory Council **ITRC**

LTM long term monitoring LTO long term operations

Missouri Department of Natural Resources **MDNR**

MS matrix spike

matrix spike duplicate **MSD**

OU operable unit

PDB passive diffusion bag PM Project Manager

PPE personal protective equipment

QAPP Quality Assurance Project Plan

quality control OC **QCP**

Quality Control Plan

SOP standard operating procedure Site Safety and Health Plan **SSHP** St. Louis Ordnance Plant **SLOP**

UFP Uniform Federal Policy

U.S. Army Corps of Engineers **USACE**

VOC volatile organic compound

DRAFT FINAL FIELD SAMPLING PLAN THE FORMER ST. LOUIS ORDNANCE PLANT ST. LOUIS, MISSOURI

REGIONAL LTO/LTM FOR SEVEN INSTALLATIONS

1.0 INTRODUCTION

This Field Sampling Plan (FSP) was prepared by HydroGeoLogic, Inc. (HGL) to define the sample locations and procedures that will be followed in the collection and analysis of environmental samples and measurements. These data are being collected for long term monitoring (LTM) activities at the former St. Louis Ordnance Plant (SLOP) in St. Louis, Missouri (Work Plan Figure 1.1). The scope of work at the former SLOP also includes routine inspection and maintenance of the monitoring wells, which is being conducted under the long term operations (LTO) component of the task order.

The objective of this FSP is to establish procedures that ensure all activities related to the project definable features of work (DFWs), meet the project specifications, and conform to the contract requirements and applicable regulations. This FSP is one of two parts of the Site-Specific Work Plan, along with the Quality Assurance Project Plan (QAPP) in Tab 2, that comprise the Sampling and Analysis Plan. These plans provide sufficient detail regarding environmental sampling activities and analytical requirements for field and laboratory work. Standard operating procedures (SOPs) and field forms are located in the FSP appendices.

1.1 PROJECT ORGANIZATION AND RESPONSIBILITIES

Section 2.0 of the Work Plan outlines of the organizational structure, roles, and responsibilities instituted to ensure that tasks and activities meet the project objectives and the specific requirements outlined in the Performance Work Statement. Brief descriptions of the roles and responsibilities of key personnel are included in Section 2.0 of the Work Plan.

Project quality assurance/quality control (QC) roles, responsibilities, and procedures are detailed in the Quality Control Plan (QCP) submitted under separate cover with the Site-Specific Work Plan. Figure 2.1 in the QCP provides a project team organizational chart that identifies key personnel, responsibilities, and lines of authority for the former SLOP. Table 2.1 of the QCP provides project points of contact. Resumes for key members of the project team are provided in Appendix B of the Project Management Plan submitted in under separate cover in conjunction with this Work Plan.

1.2 DEFINABLE FEATURES OF WORK

DFWs for the former SLOP include the following:

- Conduct Project Management activities;
- Conduct quarterly sampling of 12 monitoring wells through 2014 and annual sampling from 2015 through 2017;
- Inspect and maintain the monitoring wells;
- Manage disposal of investigation-derived waste (IDW) purge water;
- Maintain the former SLOP data in the task order LTO/LTM project database; and
- Prepare quarterly and annual reports.

The quarterly and annual groundwater sampling consists of deploying passive diffusion bag (PDB) samplers at 12 wells associated with Operable Unit (OU)-1. The 12 wells are sampled for the following volatile organic compounds (VOCs) (Conti and CH2MHill, 2012):

- Benzene
- Naphthalene
- Carbon tetrachloride
- Chloroform
- 1,1,1,2-tetrachloroethane
- 1,1,2,2-tetrachloroethane
- 1.2-dichloroethane
- 1,1,2-trichloroethane
- cis-1,2-dichloroethene
- trans-1,2-dichloroethene
- Tetrachloroethene
- Trichloroethene
- Methylene chloride
- Vinyl chloride

The LTO activities, which will be conducted concurrently with the sampling events, consist of inspection and maintenance of the monitoring wells. An annual report, including a summary of future activities and recommendations for optimization, will be prepared annually for the former SLOP. Quarterly data reports will be prepared for the first 3 quarters to present the data and sampling activities. The final quarterly report will be included in the annual report.

2.0 FIELD SAMPLING ACTIVITIES

This section presents the sampling strategy and field procedures that will be employed during LTO/LTM field activities at the former SLOP from 2013 through 2017. Specific field activities at the former SLOP include:

- Sample 12 monitoring wells quarterly (2014 only) for VOCs.
- Well maintenance including inspection, replacement of locks, and removal of vegetation.

Information provided for each sampling activity includes a brief description of the field activity, sampling method, and sampling. Laboratory methods to be used and their associated project quality objectives and measurement performance criteria are described in the QAPP. Field activities will be conducted in accordance with the SOPs provided in Appendix A.

Field sampling activities will be documented by field personnel on the sample collection field data sheets provided in Appendix B or directly into approved electronic media. The analytical requirements for monitoring well sampling are discussed within the QAPP. Replicate samples will be obtained during each sampling activity for QC analysis as described in the QAPP and within Section 4.0.

2.1 DECONTAMINATION PROCEDURES

The following two subsections present equipment and personnel decontamination procedures to be utilized for this project.

2.1.1 Equipment Decontamination

Decontamination of equipment, materials, and personnel will be performed as a safety and health measure; to avoid cross-contamination of samples submitted for chemical analysis; and to limit the migration of contaminants between work areas on the site. Decontamination will be conducted in accordance with HGL SOP 2.01, Sampling Equipment Cleaning and Decontamination (Appendix A). Decontamination will occur near the well location in an area determined by the field team leader and placed upwind of vehicle exhaust (as needed) at each sampling location.

Reusable sampling equipment (e.g., probe rods, screens, bailers, pumps, etc.) is not anticipated to be used during completion of the LTM field activities because PDB samplers will be used for sampling. Equipment intended to be used in monitoring wells for measurements, purging, or sampling (e.g., water-level probes, etc.) will be decontaminated by washing all internal and external surfaces with low-phosphate, laboratory-grade detergent followed by tap water and deionized (DI), analyte-free water rinse.

2.1.2 Personnel Decontamination

Whenever an individual comes in contact with potentially contaminated water, decontamination will be performed by washing hands and arms with soap and water at a minimum. Any other exposed portion of the body that comes in contact with potentially contaminated water will be washed with soap and water. Similar decontamination procedures will be followed before lunch breaks or when hands and arms come in contact with soil or soiled surfaces. The regional Accident Prevention Plan/Site Safety and Health Plan (SSHP) provides further details on personnel decontamination. The Site-Specific SSHP Addendum provided in TAB 3 of the Work Plan list the contaminants of concern (COCs) for the former SLOP.

2.2 SAMPLING EVENT NOTIFICATION

HGL will notify the U.S. Army Corps of Engineers, Kansas City District (CENWK) Project Manager (PM) and the former SLOP facility contact person of each upcoming sampling event by email notification at least two weeks before the sampling event begins. The CENWK PM will then notify the U.S. Environmental Protection Agency (EPA) and the Missouri Department of Natural Resources (MDNR). The notification will include the proposed sampling date, the parameters to be analyzed, and the name of the HGL contact person who will set up the sampling date appointment. The HGL contact person may contact the former SLOP facility contact person by phone or email prior to the sampling event and schedule a sampling appointment when necessary or requested.

2.3 SITE-WIDE GROUNDWATER LEVEL MONITORING

Static water levels will be collected during each sampling event. Table 2.1 details monitoring wells to be sampled, analytes, and sampling frequency. Water levels are collected on the same day and used to help depict the potentiometric surface at a point in time. Monitoring well locations are shown on Figure 2.1 and a monitoring well construction summary is provided in Table 2.2. Groundwater levels will be recorded before retrieving PDB samplers. Table 2.2 also details PDB installation depths for each well.

Groundwater level monitoring will be conducted in accordance with EPA SOP 2043, *Manual Water Level Measurements*. All water levels will be measured using an electronic water level meter on the same day. The reported depth to water will be measured from the top of the PVC riser pipe. Groundwater level measurement data and well condition notes will be recorded in a logbook and on the Observed Water Level and Well Integrity Inspection Form (Appendix B) and will be incorporated into the project database. The following information will be included on these sheets and included on the report to CENWK:

- Weather;
- Date of measurement;
- Time of measurement;
- Depth to water;
- Elevation of well monitoring points;

- Water level elevation; and
- Notes regarding condition of well.

Tables from the previous round of water level measurements will be reviewed as water levels are collected at each location to ensure the current measurements appear reasonable.

All protective casings will be locked between events or bolted down in the case of flush mounts. Locks will be replaced as needed. The Observed Water Level and Well Integrity Inspection Form contained in Appendix B will be completed when defects are noted and/or maintenance is needed at well locations. Photographs documenting well conditions shall be taken and referenced when appropriate.

If a gate was closed before entering the property, it shall be closed after entering to ensure that access to the site is limited and controlled. No lubricants shall be used on locks due to sample contamination concerns.

2.4 GROUNDWATER MONITORING WELL SAMPLING

This section addresses the groundwater monitoring activities to be conducted as part of the LTM sampling at the former SLOP.

The results of each monitoring well sampling event will be monitored. If there is a detection of any former SLOP site COC in a monitoring well where COCs have not previously been detected, CENWK will be immediately notified.

2.4.1 Location and Frequency of Samples

Figure 2.1 shows the locations of the monitoring wells at the former SLOP. The locations, frequency of samples to be collected, and method used to collect the samples will be reviewed periodically by HGL. Monitoring and Remediation Optimization System software, as discussed in the PMP, will be utilized to evaluate current and historical site data to suggest an optimization plan. Recommendations will be presented for optimization in the annual LTM report.

2.4.2 Sampling Methodology for Passive Diffusion Bag Sampling

2.4.2.1 Passive Diffusion Bag Sampling

PDB sampling technologies have been approved for use and incorporated into the LTM at the former SLOP. They allow for the collection of groundwater monitoring well samples without performing well purging. Groundwater sampling using PDBs will be in accordance with *Technical and Regulatory Guidance for Using Polyethylene Diffusion Bag Samplers to Monitor Volatile Organic Compounds in Groundwater*, which is published by the Interstate Technology Regulatory Council (ITRC, 2004), and HGL SOP 4.0 *Groundwater Sampling using Passive Diffusion Bags* (see Appendix A).

General sampling procedures that have been followed at the site are:

- Inspect the well and measure the water level in the well and record on the Observed Water Level and Well Integrity Inspection Form (Appendix B)
- Retrieve the PDB previously installed in the well by smoothly and continuously pulling the tether upwards at a rate of about 1 foot per second.
- Using the discharge tube provided with each bag, poke a hole near the handle/bottom of the PDB by pressing one end of the discharge tube firmly into the clear polyethylene membrane at a downward angle until it pierces the membrane.
- Discharge a small amount of water as waste to purge the discharge tube, and then fill the laboratory-supplied sample containers. Fill sample containers including required field quality control samples in accordance with procedures specified in the FSP.
- Collect water quality parameters and record on the PDB Field Parameter Form (Appendix B).
- Consult the well construction information (Table 2.2) to determine the depth of suspension of the PDB sampler to be deployed for the next sampling event.
- Attach the proper length of tether to the PDB sampler, rings, and weight.
- Lower the PDB into the monitoring well slowly, letting it settle to the deployment depth selected. Remove any slack from the tether and attach the tether to the bottom ring of the J-plug well cap. Log PDB deployment information on the Passive Diffusion Bag Sampling and Deployment Form (Appendix B).
- Secure the tether at the top of the well either by attaching it to the well cap or stickup protector.
- Measure and record a final water level measurement. Loosely recap the well and lock well cover padlocks.
- Allow a minimum of 2 weeks for the conditions to stabilize in the water column of the well. Leaving the PDB in the well longer will not negatively impact the sample collection process.
- Check that every line on the Field Sampling Report has been properly filled (Appendix B).
- Transfer the sample containers, the chain of custody (CoC) Record, and field sampling report to the Sample Manager.

Sample holding times and expiration are discussed in the QAPP (TAB 2 of the Work Plan).

3.0 ANALYTICAL REQUIREMENTS, SAMPLE CONTAINERS, AND PRESERVATION REQUIREMENTS

This section presents the analytical requirements, sample container and preservation requirements, and decontamination procedures for field sampling activities related to the LTM activities at the former SLOP site.

3.1 ANALYTICAL REQUIREMENTS

The QAPP provided in TAB 2 of the Work Plan lists analytical requirements, EPA-approved analytical methods, sample containers to be used, sample preservation requirements, and holding times. A subcontracted laboratory approved by CENWK will perform the required analyses for the project. The QAPP contains the laboratory certification, validation documentation, and the Laboratory Quality Assurance Plan. The laboratory will supply sample containers and add the required preservatives as listed in the QAPP prior to shipping the sample containers to the former SLOP site. Sample packaging, labeling, and shipping are discussed in Section 6. A list of analytes is presented in Worksheet #15 of the QAPP.

3.2 SAMPLE CONTAINERS AND PRESERVATION PROCEDURES

Sample containers and preservation procedures specified for all analyses are presented in the QAPP. VOC sample containers will have the hydrochloric acid preservative added to the sample containers by the container manufacturer or subcontracted laboratory prior to being shipped to the field. Proper preservation of samples will be verified by the contract laboratory. Sample bottles, containers, and preservatives will be supplied to the contractor by the contracted analytical laboratory. Sample bottles and containers will be free of target analytes, contain documentation of preservation (if applicable), and of known quality (i.e., I-Chem 200 series or equivalent) as documented by the container manufacturer.

Samples collected will be preserved according to EPA analytical method protocols established for the parameters of interest. Appropriate measures will be taken to document that requirements with respect to temperature are maintained during transport to the laboratory, and prior to login and storage at the laboratory. The procedures for sample handling, preservation, and holding time are summarized in the QAPP, and will be in accordance with the following documents:

- U.S. Army Corps of Engineers (USACE) Engineering Manual 200-1-3, *Requirements* for the Preparation of Sampling and Analysis Plans (USACE, 2001) for sample handling and preservation; and
- Test Methods for Evaluation of Solid Waste, Final Update IIIA (EPA, 1996), for sample preservation, container, and holding time requirements.

4.0 FIELD QUALITY ASSURANCE AND QUALITY CONTROL SAMPLES

A series of QC samples—including replicate samples, temperature blanks, and trip blanks—will be collected and submitted for laboratory analysis. These samples are submitted for analysis to the same subcontracted laboratory as the regular samples. Rinsate and equipment blanks will not be collected because PDB samplers are being used which not require decontamination. QC samples are analyzed for the purpose of assessing the impact of the field sampling program on the quality of the associated analytical data. The sections that follow describe the types and quantities of field QC samples to be collected.

4.1 QUALITY CONTROL DUPLICATE SAMPLES

Field duplicate samples are samples collected in the same quantity at the same time and location and under the same conditions. Theoretically, duplicate samples are representative of the parameter of interest at a given point in space and time. Field duplicates will represent at least 10 percent of the field samples collected. Field duplicates provide information regarding the reproducibility of analytical results and account for error introduced from handling, shipping, preparing, and analyzing field samples.

The data will be validated by comparing the results from the QC samples to data from the appropriate field sample to assess field sampling precision and the consistency and quality of data produced by the laboratory.

4.2 MATRIX SPIKE AND MATRIX SPIKE DUPLICATE SAMPLES

Matrix Spike/Matrix Spike Duplicate (MS/MSD) samples will represent at least 5% of the field samples collected. A field sample is split into three portions (original, MS, and MSD) and known amounts of analytes are added (spiked) into the MS and MSD portions of the sample. The analytical results of these two portions are compared to each other for reproducibility using the relative percent difference. These results are also compared against the un-spiked portion of the sample for percent recovery of the spiked analytes.

4.3 TEMPERATURE BLANKS

Temperature blanks are bottles of water packaged in each sample cooler, allowing the laboratory to determine the temperature of the shipment without disturbing the field samples. Temperature blanks are not required for this project because the laboratory uses an infrared gun to measure the temperature of a sample in each cooler.

4.4 TRIP BLANKS

Trip blanks are required only when samples are collected for analysis of VOCs. Trip blanks are prepared in the laboratory with analyte-free water and are shipped to the former SLOP with the regular sample containers. The blanks are kept unopened in the field during site sampling activities and are shipped for analysis with the project samples. Trip blanks are

designed to evaluate VOC contamination encountered during sampling, transportation, storage. One trip blank sample will be placed in each sample cooler containing samples to analyzed for VOCs, and will be analyzed with these samples for selected VOCs.	

5.0 DOCUMENTATION REQUIREMENTS

A sample is physical evidence collected from a site or facility. Due to the possible evidentiary nature of the samples collected during groundwater monitoring, a stringent program of custody procedures will be utilized to document that each sample is accounted for from the time of collection to analysis. Documentation in logbooks and CoC Records will be employed to maintain a comprehensive record of sample collection, transfer between personnel, shipment, and receipt by the laboratory.

A critical aspect of sound sample collection and analysis protocols is the maintenance of strict CoC procedures. To maintain and document sample possession, specific procedures are to be followed. A sample is considered to be in an individual's custody if the sample is:

- In the physical possession or view of the responsible party;
- Secured to prevent tampering; or
- Placed in a restricted area by the responsible party.

This section outlines the procedures that will be followed to document sample history and integrity. Additionally, EPA SOP 2420.4C, *Field Chain of Custody for Environmental Samples*, and HGL SOP 4.07, *Field Logbook Use and Maintenance* can be referenced for procedural requirements for field documentation. These SOPs are provided in Appendix A.

5.1 FIELD LOGBOOK

A logbook will be used to document all field activities and will contain sufficient data and information to reconstruct these field activities for a specific day. Pages in the logbook will be bound and numbered. All entries will be recorded legibly in indelible ink. At the end of each day, the last page will be signed and dated by the author(s) and a line drawn through the remainder of the page. At a minimum, the daily log will contain:

- The former SLOP specific sampling event on each page;
- Date and time the field work started;
- Names, titles, and organization of sampling personnel;
- Purpose of the sampling;
- Location and description of the sample and sample site;
- Date and time each sample was collected;
- Any deviations from the Work Plan;
- Meteorological conditions at the start of sampling and changes in these conditions;
- Record of any field measurements observed;
- Communication with property owners or others;
- The specific equipment used to collect any sample (serial number and description);
- The number and type of samples collected and the sample identification;
- Packaging and shipping information; and
- Sample destination.

5.2 SAMPLE DESIGNATION

Every sample collected as part of the site LTM activities will be given a unique sample designation for identification purposes. The numbering system will be coordinated with the project chemist, the database manager, and CENWK to ensure that the proposed sample identifiers are discrete. The field sample identification designation will be printed on all sample labels and will be referenced on the sample collection field sheet and in the field logbook.

A sample identification designation of up to 12 characters, consisting of three character fields, has been developed to provide a format to facilitate the former SLOP database operations.

5.2.1 Groundwater Samples

For groundwater monitoring well samples, the first 4-character field contains the site identification (e.g., SLOP), and the second character field contains the well number (e.g., MW106). The first 2 characters of the last 6-character field identifies the approximate month of the sampling event (e.g., 01 for January, 02 for February, etc.) and the last 4 digits identify the year sampled (e.g., 092014 for September 2014, 062014 for June 2014, etc.). A sampling event spanning into another month will keep the designation of the month the majority of the event was set. Sample identifications for sampling events are designated in corresponding tables. An example of the sample identification designation for a sample collected at the former SLOP for well MW-106 in June 2014 is as follows:

SLOP-MW106-062014

5.2.2 Quality Control Samples

QC samples are identified by a "2" preceding the cluster number (e.g., 2106 for MW-106). An example of the sample identification designation for a QC sample collected for well MW-106 in June 2014 is as follows:

SLOP-MW2106-062014

Trip blank sample identification will begin with "TB". The second character field contains the QC "2" sample designation and the site identification. The third character field contains the well number of one of the samples from the batch being sent to the laboratory associated with the trip blank (e.g., 106 for MW-106). The last 6-character field will identify the month and year of the sampling event. An example of the identification designation for a QC trip blank sample sent with a groundwater sample from well MW-106 in June 2014 is as follows:

TB-SLOP-MW2106-062014

The sample identification will be printed on all quality control sample labels and will be referenced on a sample collection field sheet and in the field logbook.

5.3 SAMPLE LABELS

A waterproof sample label will be attached to each sample container and completed legibly with indelible ink. The sample labels will be affixed to the sample bottle. The labels will identify the initials of the collector, date and time of sample collection, place of collection, sample number, analysis required, and preservatives added.

5.4 CUSTODY SEALS

The custody seal will be attached to the outside of the shipping container (cooler) in such a manner that the seal must be broken to allow access to the container. The following information will be entered on each custody seal in the field:

- Date sealed; and
- Sampler's signature.

5.5 CHAIN OF CUSTODY RECORDS

All sample shipments will be accompanied by the CoC Record (Appendix B) identifying its contents in accordance with EPA SOP 2420.4C, *Field Chain of Custody for Environmental Samples* found in Appendix A. This record will be used to document sample custody transfer from the sampler, to other sampling team members (if necessary), to the courier, and finally to the analytical laboratory. The CoC Record ensures that samples can be traced from the time of field collection until they are received and analyzed by the analytical laboratory.

The information required for the CoC Record includes:

- Type of sample (grab or composite) and matrix;
- Analytical method numbers and parameter names;
- Sample number;
- Signature of sampler;
- Date and time of sample collection;
- Flagging samples expected to be elevated above action levels under comments;
- Project name, location and address; and
- Signatures of persons involved in the chain of possession.

When responsibility for a group of samples changes several times, each custodian is not required to retain a copy of the CoC Record as long as the original custody record indicates that each person accepting the samples has subsequently relinquished custody appropriately. CoC Records will be completed according to the following protocol:

- The originator fills in all requested information from the sample labels.
- The originator signs the "Relinquished by" box and keeps the copy.

- The original record sheet is shipped with the samples. A plastic shipping envelope is taped to the inside of the cooler top and the remaining two copies of the CoC Record are filed with the representative sampling documents.
- The person receiving custody checks the sample label information against the custody record. He/she also checks sample condition and notes anything unusual under "Remarks" on the custody form.
- The person receiving custody signs in the adjacent "Received by" box and keeps the original.
- The date/time will be the same for both signatures, because custody must be transferred between two individuals. However, when samples are shipped via common carrier (e.g., Federal Express), the date/time will not be the same for both signatures.
- When samples are shipped via common carrier, the original custody form is shipped with the samples and the shipper (e.g. Field Sample Custodian) keeps the copy. The shipper also keeps all shipping paper, bills of lading, etc.
- In all cases, it must be readily seen that the person receiving custody has relinquished it to the next custodian.
- If samples are left unattended or a person refuses to sign, this must be documented and explained on the CoC Record.

5.6 CORRECTIONS TO FIELD DOCUMENTS

Errors on field documents will be corrected by drawing a single line through the error and entering the correct information. Errors on a field document should be corrected by the person who made the original entry, and the erroneous information should not be obliterated. All corrections will be initialed and dated.

6.0 PACKAGING, SHIPPING, AND LABELING OF SAMPLE CONTAINERS

The following three subsections describe sample packaging, shipping, and labeling practices to be utilized for this project.

6.1 PACKAGING OF SAMPLES

Samples for laboratory analysis will be placed in containers and preserved as described in the QAPP. The contractor will follow the procedures recommended by USACE in *Requirements for the Preparation of Sampling and Analysis Plans, EM 200-1-3* (USACE, 2001) for sample packaging as follows:

- Samples will be placed in appropriate containers;
- Sample containers will be placed inside a plastic bag, kept upright in the cooler, and bubble wrapped to prevent breakage;
- Trip blanks will be wrapped and placed in the same bag as the VOC vials;
- Packing material will be placed at the bottom of the cooler;
- Sample containers will be placed within a large plastic bag and the plastic bags will be placed in the waterproof metal or insulated plastic coolers;
- An additional layer of inert packing material will be placed in the cooler to partially cover the sample containers;
- Double-bagged ice packs (at least three ice packs per cooler) will be placed around the sample containers to provide uniform cooling during shipping;
- Remaining space in the cooler will be filled with a packing material to provide stability during transport;
- The CoC Record will be placed in a self-sealing polyethylene bag and taped to the inside lid of the cooler;
- The shipping container will be closed and taped shut with duct tape or strapping tape and the drain of the cooler will be shut and sealed with duct tape;
- Custody seals will be placed over the seam at the front and rear of the cooler lid and covered with clear tape; and
- The completed shipping label and any other labels (e.g., "Fragile," "This Side Up") will be placed on the top of the cooler.

Please note that hazardous materials are not anticipated and are not covered in this document.

6.2 SHIPPING CONTAINERS FOR SAMPLES

All samples sent to the laboratory for analysis will be shipped overnight via Federal Express or similar transportation provider. The packaging, labeling, and shipping of samples will follow the International Air Transport Association, Resolution 618 effective January 1, 1992.

The contractor PM (or designated representative) will contact the laboratory as necessary to inform them of incoming samples, arrival time, and special handling or analytical procedures required. Whenever feasible, samples will be delivered to the laboratory within 24 hours of sample collection.

6.3 MARKING AND LABELING OF SHIPPING CONTAINERS

Shipping labels will be clearly printed in indelible ink with the following information in unabbreviated form on a label attached to the shipping container:

- Laboratory name, address; and a contact phone number, and
- Return name, address, and contact phone number.

The names, addresses, and phone numbers of the analytical laboratories are presented in Worksheet #6 of the QAPP.

7.0 INVESTIGATION-DERIVED WASTES

IDW from groundwater monitoring activities may consist of water generated from sampling, used personal protective equipment (PPE), and disposable sampling equipment. IDW will be managed in accordance with *Management of IDW During Site Inspections* (EPA, 1991) and other applicable guidance.

PDBs will be used; therefore, purge water will not be generated from sampling activities. However, a minimal amount of sample material may remain in the PDB after the sample bottles have been filled. Any remaining water will be containerized. Aqueous waste will be transferred into a U.S. Department of Transportation–approved 55-gallon drum, characterized, and discharged into the on-site combined sanitary and stormwater sewer (following Metropolitan Sewer District approval).

Solid waste (PPE and disposable sampling equipment) will be stored in trash bags and will be properly disposed of at the end of each sampling event. Measures will be taken to control the generation of excess waste. IDW generated during project is expected to be nonhazardous; therefore, county landfill facilities will be utilized for the disposal of such items including garbage, debris, used PPE, etc.

8.0 CHEMICAL QUALITY CONTROL

Chemical QC consists of three phases (preparatory, initial, and follow-up) of control that will be performed for the sampling activities.

8.1 PREPARATORY PHASE

The contractor PM and the sampling team will discuss the requirements for the project as described in Section 2. Each sampler will be given or have access to a copy of the Work Plan. Each sampler also will be given a copy of the current year's sampling tables and figures. The preparatory meeting will include a discussion of the following:

- Review of the types and locations of media to be sampled;
- Review of site for chemical, biological, and physical hazards;
- Review of the PPE required during sampling;
- Review of the groundwater level measurement procedures (where applicable);
- Review of the sampling techniques to be used;
- Examination of the required sampling equipment and materials;
- Review of the decontamination procedures; and
- Review of the sample documentation, packaging, shipping, and labeling requirements.

This phase will be repeated for new sampling personnel added to the team.

8.2 INITIAL PHASE

During the initial phase, the PM will supervise the initial sampling activities to ensure the required sampling procedures are followed.

8.3 FOLLOW-UP PHASE

The follow-up phase requires that the PM review the progress of sampling activities to ensure compliance with all sampling methods. Supervision of the sample packaging, shipping, and labeling, as described in Section 6, is part of the follow-up phase of chemical QC.

9.0 CORRECTIVE ACTIONS

Any field sampling problems or deficiencies (e.g., improper sampling, decontamination, or packaging procedures) detected during the initial or follow-up phases of quality control will be documented and corrected immediately. After corrective actions are taken, the follow-up phase of quality control will be intensified until the PM is satisfied that the problem is corrected. CENWK will be notified as soon as possible concerning sampling problems or deficiencies and any corrective actions taken. The information also will be maintained in the project files.

10.0 REFERENCES

- Conti and CH2MHill, 2012. Final Long-Term Management/Land Use Control Implementation Plan Operable Unit 1St. Louis Ordnance Plant Former Hanley Area, St. Louis, Missouri. September
- Interstate Technology Regulatory Council (ITRC), 2004. *Technical and Regulatory Guidance for Using Polyethylene Diffusion Bag Samplers to Monitor Volatile Organic Compounds in Groundwater*. February.
- U.S. Environmental Protection Agency (EPA), 1991. Management of IDW During Site Inspections
- EPA, 1996. Test Methods for Evaluating Solid Waste (SW-846). Third Edition. Final Update IIIA. December.
- USACE, 2001. Requirements for Preparation of Sampling and Analysis Plans. Engineer Manual No. 200-1-3. February.



Table 2.1
Sample Summary
The Former St. Louis Ordnance Plant

(Se			.		20	13			20	14			20	15			20	16			20	17	
Subsite (sample analyses)	Well Location	Top Screen Depth (ft bgs)	Bottom Screen Depth (ft bgs)	10	20	3Q	40	10	20	30	40	10	20	30	40	10	20	30	40	10	2Q	30	40
	MW-106	15	35				X	X	X	X	X		X				X				X		
	MW-107	10	27				X	X	X	X	X		X				X				X		
	MW-108	10	27				X	X	X	X	X		X				X				X		
	MW-109	10	28				X	X	X	X	X		X				X				X		
	MW-110	10	28				X	X	X	X	X		X				X				X		
OU-1	MW-112	10	28				X	X	X	X	X		X				X				X		
(VOCs)	MW-113	10	27				X	X	X	X	X		X				X				X		
	MW-114	9	29				X	X	X	X	X		X				X				X		
	MW-115	33	43				X	X	X	X	X		X				X				X		
	MW-116	18	28				X	X	X	X	X		X				X				X		
	MW-118	26	36				X	X	X	X	X		X				X				X		
	MW-119	10	30				X	X	X	X	X		X				X				X		

Notes:

bgs = below ground surface

ft = feet

VOC = volatile organic compound

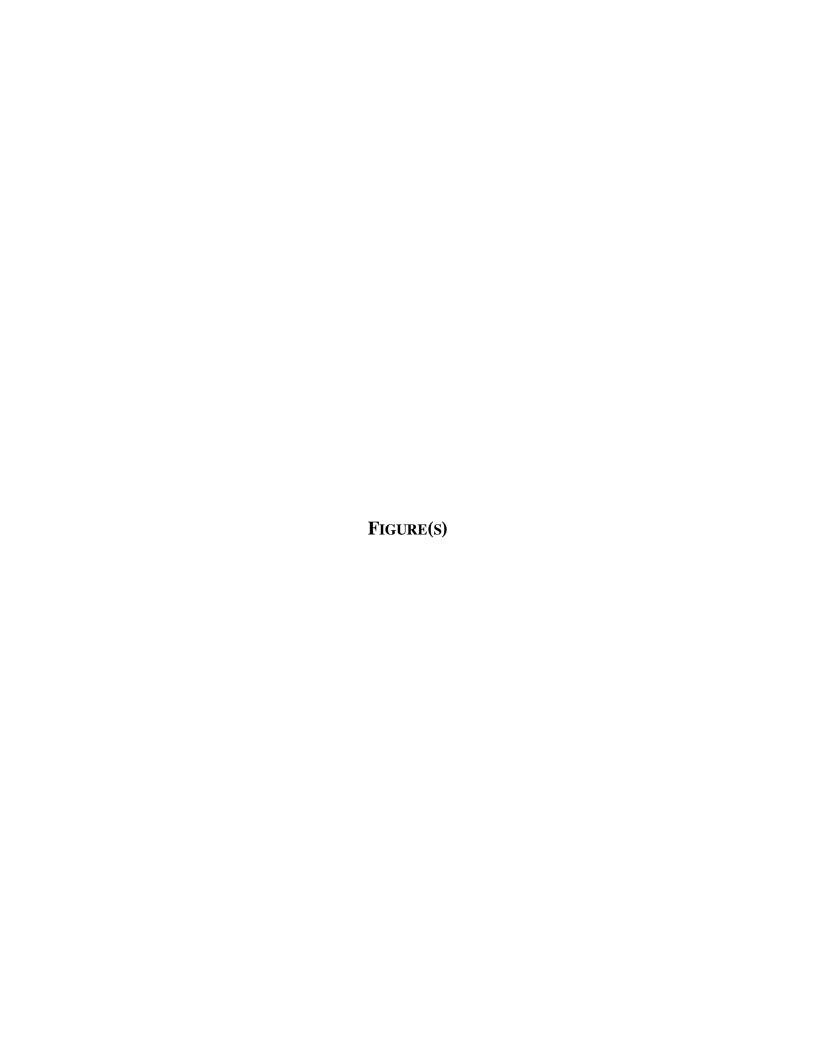
Table 2.2 Monitoring Well Summary The Former St. Louis Ordnance Plant

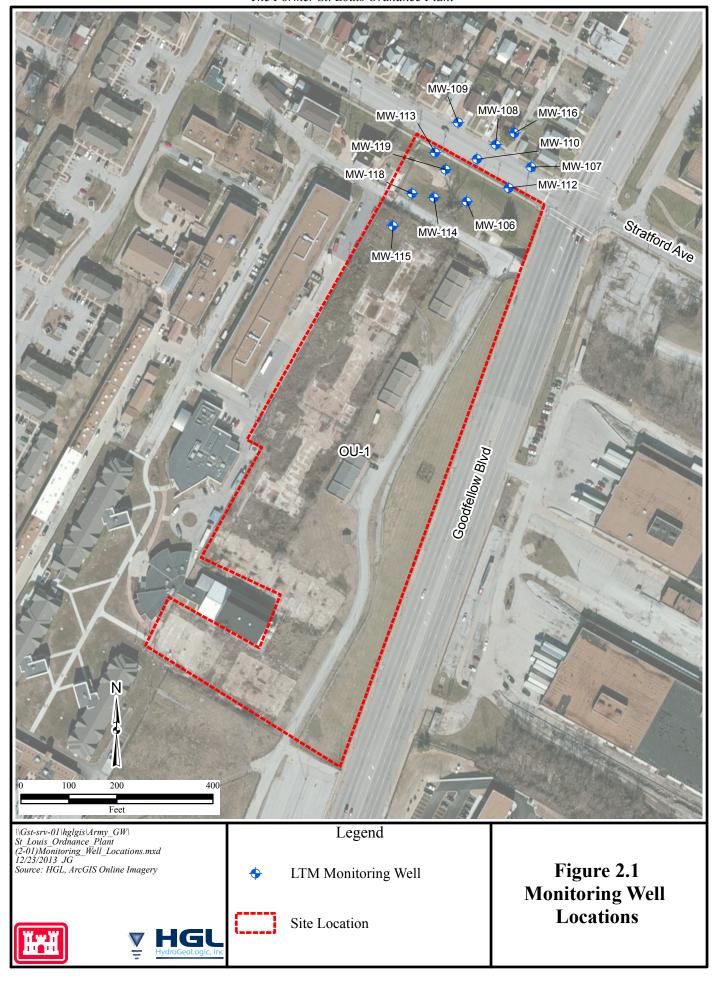
Monitoring Well ID	Date Installed	Well Diameter (inches)	Total Depth (ft bgs)	Screened Interval (ft bgs)	Passive Diffusion Bag Installation Depth ^a (ft bgs)
MW-106	1/22/2005	2	35	15.0-35.0	25
MW-107	1/25/2007	2	27	10.0-27.0	18.5
MW-108	1/25/2007	2	27	10.0-27.0	18.5
MW-109	1/26/2007	2	28	10.0-28.0	19
MW-110	1/25/2007	2	28	10.0-28.0	19 ^b
MW-112	1/25/2007	2	28	10.0-28.0	19
MW-113	1/26/2007	2	27	10.0-27.0	18.5
MW-114	3/20/2007	2	29	9.0-29.0	19
MW-115	5/19/2008	2	43	33.0-43.0	38 ^c
MW-116	5/16/2008	2	28	18.0-28.0	23
MW-118	8/11/2010	2	36	26.0-36.0	31
MW-119	5/9/2012	2	30	10.0-30.0	20

^aInstall the midpoint of the PDB at the midpoint of the screened interval.

^bThe PDB may have to be installed at 12 ft bgs due to an apparent obstruction in the well.

^cThis well is a stick-up well that rises roughly 3 feet above ground surface level. Therefore, the length of the cable is approximately 41 feet long.





APPENDIX A

STANDARD OPERATING PROCEDURES

- HGL SOP 2.01, Sampling Equipment Cleaning and Decontamination
- EPA SOP 2043, Manual Water Level Measurements
- HGL SOP 4.0 (Interim) Groundwater Sampling using Passive Diffusion Bags
- EPA SOP 2420.4C, Field Chain of Custody for Environmental Samples
- HGL SOP 4.07, Field Logbook Use and Maintenance



STANDARD OPERATING PROCEDURE

Sampling Equipment Cleaning and Decontamination

SOP No.: 2.01

SOP Category: HTRW

Revision No.: 1
Date: December 2010

1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to describe decontamination methods and related issues involving the physical process of removing chemical and radioactive contaminants from sampling equipment.

2.0 SCOPE AND APPLICATIONS

This procedure is specifically applicable to decontaminating the surfaces of sampling equipment that come in direct contact with actual samples during sample collection and processing. This SOP describes the procedures to be followed to achieve effective decontamination as follows: (1) remove contaminants from contaminated surfaces, (2) minimize the spread of contamination to uncontaminated surfaces, (3) avoid any cross-contamination of samples, and (4) minimize personnel exposures. The intent is to accomplish the required level of decontamination while minimizing the generation of additional solid and liquid waste.

Other decontamination procedures may apply to a specific project; refer to the work plan for project-specific decontamination methods and schedules.

3.0 GENERAL REQUIREMENTS

All work will be performed in a manner that is consistent with Occupational Safety and Health Administration established standards and requirements. Refer to the site- or project-specific health and safety plan for relevant health and safety requirements. All activities will be conducted in conformance with the Site Health and Safety Plan. Procedures for packaging and disposing of all waste generated during field activities will be described in the project-specific work plan.

Personnel who use this procedure must provide documented evidence to the program manager or project manager that they have been trained on the procedure. This documentation will be retained in the project file.

Any deviations from specified requirements will be justified to and authorized by the project manager and/or the relevant program manager and discussed in the approved project plans. Deviations from requirements will be sufficiently documented to re-create the modified process.

4.0 **DEFINITIONS**

Deionized Water: Tap water treated by passing through a standard deionizing resin column. The deionized water should contain no heavy metals or other inorganic compounds (in other words,

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compounds at or above analytical detection limits) as defined by a standard inductively coupled argon plasma spectrophotometer scan.

Equipment: Those items (variously referred to as "field equipment" or "sampling equipment") necessary to conduct sampling activities, but that do not directly contact the samples.

Laboratory Detergent: A standard brand of phosphate-free laboratory detergent, such as Liquinox®, or the equivalent.

Organic-Free Water: Tap water treated with activated carbon and deionizing units or water from a Milli-Q® system (or equivalent). This water should not contain pesticides, herbicides, extractable organic compounds, and less than 50 micrograms per liter of purgeable organic compounds as measured by a low-level gas chromatography/mass spectrometry scan. Organic-free water should be stored only in glass or Teflon® containers and dispensed from only glass, Teflon, or stainless steel containers.

Sampling Devices: Utensils and other implements that come into direct contact with samples during their collection and processing.

Solvent: Substance capable of dissolving other substances. Pesticide-grade isopropanol is the most common solvent used to decontaminate equipment. Using any other solvent must be justified and approved by the responsible project personnel and documented on the Daily Field Report forms or in the field logbooks.

Tap Water: Water from a tested and approved water system.

5.0 PROCEDURES

5.1 GENERAL

Decontamination of sampling devices will be performed in a designated decontamination area, removed from any sampling location. This designated area must also be in a location free of direct exposure to airborne and radiological surface contaminants, and downwind of the location where clean field equipment, clean sample devices, and sample containers are stored.

As a minimum, nitrile or equivalent gloves will be worn while decontaminating equipment. Safety glasses or goggles, uncoated Tyvek® coveralls, laboratory coat, or splash apron will be worn if justified by the contaminant concentration and potential adverse effects. If cleaning with steam or high-temperature water, a face shield, heavy-duty polyvinyl chloride (PVC) or equivalent gloves, coated Tyvek or equivalent coveralls will be worn. Ground-fault circuit interrupters will be used to supply power to any portable electrical equipment in the equipment decontamination area. Solvent rinsing will be conducted in an open, well-ventilated area or under a fume hood. No eating, smoking, drinking, chewing, or hand-to-mouth contact will be permitted during

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decontamination activities. A 15-minute eyewash will be available within 100 feet if corrosive (concentrated acids or bases) decontamination fluids are used.

- Contaminated or dirty sampling devices/equipment should not be stored with clean (decontaminated) sampling devices/equipment.
- Clean, decontaminated sampling devices should be segregated from all other equipment and supplies.
- Paint or any other coatings must be removed from any part of a sampling device that may either contact a sample or may otherwise affect sample integrity. After such coatings are removed, the sampling device will then require decontamination by the appropriate method.
- The brushes used to clean sampling devices must not be of the wire-wrapped type.
- For any of the specific decontamination methods that may be used, the substitution of higher-grade water is permitted (for example, using organic-free water in place of deionized water). However, it must be noted that deionized water and organic-free water are less effective than tap water in rinsing away the detergent during the initial rinse.
- Decontaminated sampling devices and all filled and empty sample containers will be stored in locations that are protected from exposure to any contaminant.
- The method for decontaminating sampling devices and the exterior of sample containers that have been exposed to radioactive material is based on the material contaminated, the sample medium, the radiation levels, and the specific radionuclides to be removed.
- The release of decontaminated sampling devices and sample containers for unrestricted use is based on site-specific criteria. These site-specific criteria should be detailed in the project-specific work plan.
- Rags used during decontamination activities may become a hazardous waste and require segregation. Refer to the project work plans for hazardous waste disposal requirements.

5.2 DECONTAMINATION SCHEDULES

- Sampling devices must be decontaminated before being used in the field to prevent potential contamination of a sample.
- Sampling devices must be decontaminated between samples to prevent crosscontamination.
- Sampling devices must be decontaminated at the close of the sampling event before being taken off site.

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An acceptable alternative to cleaning and decontaminating sampling devices is
using items cleaned or sterilized by the manufacturer that are discarded after use.
Care must be exercised to ensure such previously cleaned or sterilized items do not
retain residues of chemical or radioactive sterilizing agents that might interfere
with analytical techniques.

- Whenever visible dirt, droplets of liquid, stains, or other extraneous materials are detected on the exterior of a sample container, the exterior surfaces must be decontaminated. This step should be performed before the container is placed in a sample cooler or shipping container.
- For sample containers used in controlled access areas, a more rigorous cleaning and/or radiation monitoring may be required before removal from the site. Refer to the project-specific work plan for details.

5.3 DECONTAMINATION METHODS

The following decontamination methods are examples of some of those most commonly used in field investigations. Note that the decontamination methods described in this section are for guidance only; the field operations manager will adjust decontamination practices to fit the sampling situation and applicable requirements.

- The exterior of sample containers This decontamination will be performed at the sample location before the sample container is placed in the sample cooler or shipping container as follows:
 - The exterior surfaces of the sample container must be wiped with disposable rags/toweling, or rinsed with deionized water.
 - o If rinsing with deionized water, the exterior of the sample container must be wiped dry with disposable rags/toweling, or allowed to air dry.
 - o All visible dirt, droplets of liquid, or other extraneous materials must be removed.
 - For containers used in controlled-access areas or where the sample media are difficult to remove (for example, sludge), a more rigorous cleaning and/or radiation monitoring may be required. Refer to the project-specific work plan for details.
- Decontaminating stainless steel, Teflon, or metal sampling devices used to collect samples for trace organic compounds and/or metals analyses:
 - Clean with a tap water and laboratory detergent solution. Use phosphatefree detergent, such as Liquinox or equivalent. Use a brush to remove particulate matter and surface film.
 - o Rinse thoroughly with organic-free water.

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- o Rinse twice with solvent (pesticide-grade isopropanol).
- o Allow to air dry for 24 hours, if possible.
- o If it is not possible to air dry for 24 hours, then rinse twice with organic-free water and allow to air dry as long as possible.
- Wrap sampling devices with aluminum foil (with shiny side facing outward).

Note: When a sampling device is used to collect samples that contain oil, grease, or other hard-to-remove materials, it may be necessary to rinse the device several times with an approved solvent (one that meets the requirements of the work plan) before initiating decontamination. In extreme cases, it may be necessary to steam clean, brush, or sandblast the sampling device before using this decontamination method. If the sampling device cannot be adequately cleaned using the above means, it must be discarded.

- Decontaminating glass sampling devices used for the collection of samples for trace organic compounds and/or metals analyses
 - Wash thoroughly with laboratory detergent and hot water using a brush to remove any particulate matter or surface film.
 - o Rinse thoroughly with hot tap water.
 - o Rinse thoroughly with tap water.
 - o Rinse twice with solvent and allow to air dry for at least 24 hours, if possible.
 - Wrap with aluminum foil (shiny side facing outward) to prevent contamination during storage and/or transport to the field.

Note: When a sampling device is used to collect samples that contain oil, grease, or other hard-to-remove materials, it may be necessary to rinse the device several times with an approved solvent (one that meets the requirements of the work plan) before initiating decontamination. In extreme cases, it may be necessary to steam clean, brush, or sandblast the sampling device before using this decontamination method. If the sampling device cannot be adequately cleaned using the above means, it must be discarded.

5.4 QUALITY CONTROL

The quality of the deionized and organic-free water used may be monitored by collecting samples in standard precleaned sample containers and submitting them to the laboratory for a standard inductively coupled plasma scan. Organic-free water should be submitted for low-level pesticide, herbicide, extractable, or purgeable compounds analyses, as appropriate.

SOP No.: 2.01

SOP Category: HTRW

Revision No.: 1
Date: December 2010

The effectiveness of the decontamination procedures is monitored by submitting samples of rinse water to the laboratory for low-level analyses of the parameters of interest. An attempt should be made to select different sampling devices each time devices are decontaminated to ensure a representative sampling of all devices is obtained over the length of the project. Note on the Daily Field Report Form or in the field logbooks the devices being used for the rinsate samples.

6.0 RECORDS

Documentation generated as a result of this procedure is collected and maintained in accordance with requirements specified in the work plan.

SOP No.: 2.01

SOP Category: HTRW

Revision No.: 1

Date: December 2010

ATTACHMENTS

Below is Attachment 1, Field Checklist

ATTACHMENT 1 FIELD CHECKLIST	
Daily Field Report Forms or Field Logbooks	Gloves
Safety Glasses or Monogoggles	Safety Shoes
Black, Indelible Pen	Plastic Sheeting
Decontamination Equipment	Health and Safety Plan
Work Plan	Monitoring Instruments
Appropriate Containers for Waste and Equipment	



STANDARD OPERATING PROCEDURES

SOP: 2043 PAGE: 1 of 10 REV: 0.0 DATE: 02/11/00

MANUAL WATER LEVEL MEASUREMENTS

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- 3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE
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- 8.0 CALCULATIONS
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- 11.0 HEALTH AND SAFETY
- 12.0 REFERENCES
- 13.0 APPENDIX
 - A Groundwater Level Data Form

SUPERCEDES: SOP #2043; Revision 0.0; 10/03/94; U.S. EPA Contract 68-C4-0022.



STANDARD OPERATING PROCEDURES

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MANUAL WATER LEVEL MEASUREMENTS

1.0 SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to set guidelines for the determination of the depth to water and separate phase chemical product (i.e., gasoline, oil, PCE, TCE) in an open borehole, cased borehole, monitor well, or piezometer. These standard operating procedures may be varied or changed as required, dependent on site conditions, and equipment limitations. In all instances, the actual procedures employed will be documented and described in an appropriate site report. Mention of trade names or commercial products does not constitute U.S. EPA endorsement or recommendation for use.

Generally, water-level measurements taken in boreholes, piezometers, or monitor wells are used to construct water table or potentiometric surface maps and to determine flow direction as well as other aquifer characteristics. Therefore, all water level measurements at a given site should preferably be collected within a 24 hour period. However, certain situations may produce rapidly changing groundwater levels that necessitate taking measurements as close in time as possible. Large changes in water levels among wells may be indicative of such a condition . Rapid groundwater level changes may occur due to:

- ! Atmospheric pressure changes
- ! Tidal influences
- ! Changes in river stage, impoundments levels, or flow in unlined ditches
- ! Pumping of nearby wells
- ! Precipitation

2.0 METHOD SUMMARY

A survey mark should be placed on the top of the riser pipe or casing as a reference point for groundwater level measurements. If the lip of the riser pipe is not flat, the reference point may be located on the grout apron or the top of the outer protective casing (if present). The measurement reference point should be documented in the site logbook and on the groundwater level data form (Appendix A), if used. All field personnel must be made aware of the measurement reference point being used in order to ensure the collection of comparable data.

Before measurements are made, water levels in piezometers and monitor wells should be allowed to stabilize for a minimum of 24 hours after well construction and development. In low yield situations, recovery of water levels to equilibrium may take longer. All measurements should be made to an accuracy of 0.01 feet. Water level measuring equipment must be decontaminated and, in general, measurements should proceed from the least to the most contaminated wells.



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MANUAL WATER LEVEL MEASUREMENTS

Open the well and monitor the headspace with the appropriate air monitoring instrument to determine the presence of volatile organic compounds. For electrical sounders lower the device into the well until the water surface is reached as indicated by a tone or meter deflection. Record the distance from the water surface to the reference point. Measurement with a chalked tape will necessitate lowering the tape below the water level and holding a convenient foot marker at the reference point. Record both the water level as indicated on the chalked tape section and the depth mark held at the reference point. The depth to water is the difference between the two readings. Remove measuring device, replace riser pipe cap, and decontaminate equipment as necessary. Note that if a separate phase is present, an oil/water indicator probe is required for measurement of product thickness and water level.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

This section is not applicable to this standard operating procedure (SOP).

4.0 POTENTIAL PROBLEMS

- 1. Cascading water, particularly in open-hole or rock wells, may interfere with the measurement.
- 2. Some older types of electric sounders are only marked at five-foot intervals. A surveyor's tape is necessary to extrapolate between the 5-foot marks.
- 3. Oil or other product floating on the water column can insulate the contacts of the probe on an electric sounder and give false readings. For accurate level measurements in wells containing floating product, a special oil/water level indicator is required.
- 4. Tapes (electrical or surveyor's) may have damaged or missing sections, or may be spliced inaccurately.
- 5. An airline may be the only available means to make measurements in sealed production wells but the method is generally accurate only to approximately 0.2 foot.
- 6. When using a steel tape, it is necessary to lower the tape below the water level in order to make a measurement. This assumes knowledge of the approximate groundwater level.

5.0 EQUIPMENT

The electric water level indicator and the chalked steel tape are the devices commonly used to measure water levels. Both have an accuracy of 0.01 feet. Other field equipment may include:

Air monitoring instrumentation



STANDARD OPERATING PROCEDURES

SOP: 2043 PAGE: 4 of 10 REV: 0.0 DATE: 02/11/00

MANUAL WATER LEVEL MEASUREMENTS

- Well depth measurement device
- Chalk
- Ruler
- Site logbook
- Paper towels and trash bags
- Decontamination supplies as outlined in Section 7.2 or the current approved site specific work plan
- Groundwater level data forms

6.0 REAGENTS

No chemical reagents are used in this procedure; however, decontamination solutions may be necessary. If decontamination of equipment is required, refer to ERT/REAC SOP #2006 Rev 0.0 08/11/94, *Sampling Equipment Decontamination*, and the current approved site specific work plan.

7.0 PROCEDURES

7.1 Preparation

- 1. Determine the number of measurements needed, the methods to be employed, and the equipment and supplies needed.
- 2. Decontaminate or pre-clean equipment, and ensure that it is in working order.
- 3. Coordinate schedule with staff, clients, and regulatory agency, if appropriate.
- 4. If this is an initial visit, perform a general site survey prior to site entry in accordance with the current approved site specific Health and Safety Plan.
- 5. Identify sampling locations.



STANDARD OPERATING PROCEDURES

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MANUAL WATER LEVEL MEASUREMENTS

7.2 Procedures

Procedures for determining water levels are as follows:

- 1. If possible, and when applicable, start at those wells that are least contaminated and proceed to those wells that are most contaminated.
- 2. Clean all the equipment entering the well(s) by the following decontamination procedure:
 - Triple rinse equipment with deionized water.
 - Wash equipment with an Alconox solution which is followed by a deionized water rinse.
 - Rinse with an approved solvent (e.g., methanol, isopropyl alcohol, acetone) as per the work plan, if organic contamination is suspected.
 - Place equipment on clean surface such as a teflon or polyethylene sheet to air dry.
- 3. Remove locking well cap, note well ID, time of day, and date in site logbook or an appropriate groundwater level data form.
- 4. Remove well cap.
- 5. If required by site-specific condition, monitor headspace of well with a photoionization detector (PID) or flame ionization detector (FID) to determine presence of volatile organic compounds, and record results in site logbook.
- 7. Lower water-level measuring device into the well. Electrical tapes are lowered to the water surface whereas chalked steel tapes are lowered generally a foot or more below the water surface. Steel tapes are generally chalked so that a 1-to 5-foot long section will fall below the expected water level.
- 8. For electrical tapes record the distance from the water surface, as determined by the audio signal or meter, to the reference measuring point and record in the site logbook. For chalked tapes, an even foot mark is held at the reference point, once the chalked section of the tape is below the water level. Both the water level on the tape and the foot mark held at the reference point is recorded. The depth to the water is then the difference between the two readings. In addition, note the reference point used (top of the outer casing, top of the riser pipe, ground surface, or some other reproducible position



STANDARD OPERATING PROCEDURES

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MANUAL WATER LEVEL MEASUREMENTS

on the well head). Repeat the measurement.

- 9. Remove all downhole equipment, replace well cap and locking steel caps.
- 10. Rinse all downhole equipment and store for transport to the next well. Decontaminate all equipment as outlined in Step 2 above.
- 11. Note any physical changes, such as erosion or cracks in protective concrete pad or variation in total depth of well, in field logbook or on groundwater level data form.

8.0 CALCULATIONS

To determine groundwater elevation above mean sea level, use the following equation:

$$E_W = E - D$$

where:

 $E_{\rm w}$ = Elevation of water above mean sea level (feet) or local datum

E = Elevation above sea level or local datum at point of measurement (feet)

D = Depth to water (feet)

9.0 QUALITY ASSURANCE/QUALITY CONTROL

The following general quality assurance/quality control (QA/QC) procedures apply:

- 1. All data must be documented on field data sheets, groundwater level data forms, or within personal or site logbooks.
- 2. All instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the work plan.
- 3. Each well should be tested at least twice in order to compare results. If results do not agree to within 0.02 feet, a third measurement should be taken and the readings averaged. Consistent failure of consecutive readings to agree suggests that levels are changing because of one or more conditions as indicated in Section 1.

10.0 DATA VALIDATION

This section is not applicable to this SOP.



STANDARD OPERATING PROCEDURES

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MANUAL WATER LEVEL MEASUREMENTS

11.0 HEALTH AND SAFETY

The results of monitoring the well head and breathing zone with a FID or PID, as per section 7.2, may indicate the need to upgrade the personal protection level according to the current approved site Health and Safety Plan.

12.0 REFERENCES

Driscoll, F.G. 1986. Groundwater and Wells. Second Edition. Chapter 16. *Collection and Analysis of Pumping Test Data.* pp 534-579. Johnson Filtration Systems Inc. St. Paul, Minnesota.

- U.S. Environmental Protection Agency, 1986. RCRA Groundwater Monitoring Technical Enforcement Guidance Document, pp. 207.
- U.S. Environmental Protection Agency, 1987, A Compendium of Superfund Field Operations Methods. EPA/540/p-87/001 Office of Emergency and Remedial Response Washington, D.C. 20460.



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MANUAL WATER LEVEL MEASUREMENTS

APPENDIX A Groundwater Level Data Form SOP #2043 February 2000



Other significant observations:

U. S. EPA ENVIRONMENTAL RESPONSE TEAM

STANDARD OPERATING PROCEDURES

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MANUAL WATER LEVEL MEASUREMENTS

						PAGE C
ITE NAME	:				LOGGER NA	AME:
OG DATE:					WBS #: <u>RIA</u>	
	<u> </u>	T	T		-	
Well I.D.	Time	Elevation of well ⁽¹⁾ (T.O.C.)	Depth to bottom of well (ft)	Depth to water (ft)	Depth to product (ft)	COMMENTS (pH, temperature, specific conductance)
TO	C: top of ca	asing	(1) feet above	maan saa la	uol	



EPA AES Contract Standard Operating Procedure:

Groundwater Sampling using Passive Diffusion Bags

Category: SOP No. 4.0 (Interim)
QA/QC Date: December 2008

1.0 INTRODUCTION

Use of passive diffusion bags (PDBs) in collecting groundwater samples from monitoring wells allows groundwater samples to be collected without inducing flow from the well, which eliminates the time required for well purging and the need for handling and disposal of the purged water. Use of PDBs enables the collection of groundwater samples for volatile organic compound (VOC) analysis with minimal agitation of the water being sampled and provides comparable results to low-flow groundwater sampling methods. Multiple PDBs can be used to target specific, multiple intervals within a well, or to collect additional sample volume. Further, use of PDBs eliminates the need to collect field readings of pH, conductivity, temperature, oxidation-reduction potential (ORP), dissolved oxygen, and turbidity, as these well parameters do not need to stabilize before the sample is retrieved.

2.0 PURPOSE

The purpose of this SOP is to establish general reference information for sampling groundwater using PDBs. The methods detailed in this SOP are not inclusive of all conditions that may be encountered in the field that may affect sample collection techniques using PBDs. The sampling and data collection procedures described in this SOP are designed to be used concurrently with laboratory analysis for common types of groundwater contaminants, such as VOCs, semivolatile organic compounds (SVOCs), pesticides, metals, and some biological parameters. In all instances, the specific procedure employed for sampling at a site should be recorded in the field logbook and detailed in the final report. The reference of specific trade names or commercial products does not constitute endorsement or recommendation for use.

3.0 PDB SAMPLE COLLECTION PROCEDURES

The following materials and equipment are needed to support PDB sampling activities at each selected well:

- One 350 or 500-milliliter (mL) PDB
- Discharge tube
- One or two 20 oz. weights (based on deployment depth)
- Nylon zip ties (2 per PDB)
- One locking J-plug well cap with ring
- 3/16-inch poly tether (based on depth of bag deployment)
- Deionized or distilled water for installation of the PDBs

A diagram and description of the PDB sampling equipment purchased from EON Products, Inc. (EON) are provided as Attachment 1.

Based upon the manufacturer's specification for the PDB, the PDB will diffuse groundwater from the center point of the device for 2.5 feet vertically in each direction. Therefore, the center of the sampler should be placed no deeper than 2.5 feet from the bottom of the screen. Monitoring for light, non-aqueous phase liquids (NAPLs) or dense NAPLs may require additional assessment and evaluation for deployment depth if free product has been observed in the well.

3.1 Deployment of Passive Diffusion Bags into Monitoring Wells

The standard deployment procedures for PDBs are summarized below:

- Collect a field blank of the deionized or distilled water used for PDB installation. If the water includes a certificate of purity from the supplier, or is of known quality, then this step can be skipped.
- Using the well-specific deployment depth (center of screen or other), measure that distance plus two feet of poly tether and cut to length. Depending on the supplier, the poly tether may come from the supplier pre-cut to the appropriate length.
- Measure up approximately two inches on one end of the poly tether. Using the ring on the weight, slide through the tether to attach the weight. Go up one inch above the top of the weight, and repeat previous steps with second weight, if required. Depending on the supplier, the tether, ring, and weights may come pre-assembled for the PDBs.
- Remove PDB from shipping bag, take off cap, and using fill kit, fill PDB up to top making a "crown" on lip. Squeeze the sampler several times and add more water. Repeat as needed to expand the membrane and remove air pockets. PDB bags may come pre-filled if ordered that way. If the bags come pre-filled, skip this step.
- Insert the plug firmly into the PDB, until the rim of the plug is as close to the nozzle as possible.
- Place a zip tie through the poly tether and attach to bottom ring on the PDB. Make sure the zip tie is snug.
- Going above the membrane bag of the PDB, run a zip tie through the protective mesh screen making a loop the size of a dime. Take another zip tie and run it through the poly tether and through the first zip tie just attached to the top of the PDB, and pull snug.
- Cut off any excess lengths of zip tie left over to prevent snags.
- Lower the PDB into the monitoring well slowly, letting it settle to the deployment depth selected. Remove any slack from the tether and attach the tether to the bottom ring of the J-plug well cap. Use another zip tie to attach the J-plug well cap to the tether.
- Once the PDB has been deployed, record the required information on the revised Groundwater Field Sampling Data Sheet and field logbook, including date and time of bag deployment. Based upon the contaminants of concern and the type of PDB, record the minimum equilibration time required before retrieval of the PDB. For VOCs, the

- standard deployment time is a minimum of two weeks. Leaving the PDB in the well longer will not negatively impact the sample collection process.
- It should be noted that one 350-ml PDB is more than sufficient to fill four 40-ml vials required by the EPA laboratory for each VOC sample. If additional volume is required for splits, matrix spike/matrix spike duplicates (MS/MSDs) or field duplicates, a second PDB should be deployed within the well to ensure sufficient sample volume is collected. Multiple bags should be attached sequentially on the poly tether.

3.2 Pre-Sampling Procedures

Sampling of monitoring wells should be performed starting with the least contaminated wells and moving to the most contaminated wells. Before accessing the well for sampling, the sampler shall observe the condition of the monitoring well and record observations on the HGL Groundwater Field Sampling Data Sheet. Then remove the cap from the well and allow sufficient time for the groundwater level to equilibrate with atmospheric conditions. The depth to groundwater shall be measured and recorded (to 0.10 inch) on the HGL Groundwater Field Sampling Data Sheet. After the water level has been recorded, the sampler shall decontaminate the water level indicator/interface probe per procedures specified in the SAP.

3.3 PDB Retrieval and Sampling Procedures

All bottleware used to containerize VOC samples shall be pre-cleaned, traceable to the facility, and prepreserved. The standard for retrieving PDBs fro monitoring wells and containerizing the VOC samples are summarized below:

- With minimal disturbance of the groundwater, remove the PDB(s) from the well.
- Carefully remove the zip ties used to hold PDB(s) to the tether using scissors or a box cutter, being careful not to puncture the bag.
- Using the discharge tube provided with each bag, poke a hole near the handle/bottom of the PDB by pressing one end of the discharge tube firmly into the clear polyethylene membrane at a downward angle until it pierces the membrane.
- Discharge a small amount of water as waste to purge the discharge tube, then fill the laboratory-supplied sample containers. Fill sample containers including required field quality control samples in accordance with procedures specified in the SAP.
- Following sample collection, discharge any remaining groundwater in the PDB into a bucket for disposal in the local sanitary sewer.
- If the sample labels are not preprinted, complete the sample labels and attach the labels to the containers. Record the location, date, and time of the sample collection in the field logbook and on the Groundwater Field Sampling Data Sheet.
- Store samples in coolers with ice and complete the chain-of-custody record as specified in the SAP.
- Containerize the used PDB in a garbage bag for disposal.

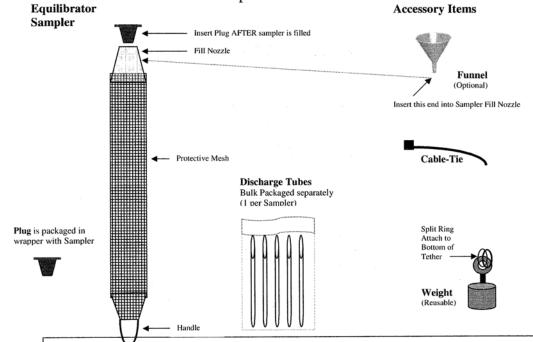
These procedures shall be repeated for each monitoring well to be sampled using PDBs. If periodic (e.g., quarterly long term monitoring [LTM]) sampling is to occur at particular wells, deploy the PDB for the subsequent round of sampling. This eliminates a second trip to the site for PDB installation.

ATTACHMENT 1

PDB Instructions – EON Products, Inc. Equilibrator Diffusion Sampler



EQUILIBRATOR TM **Diffusion Sampler Instructions**



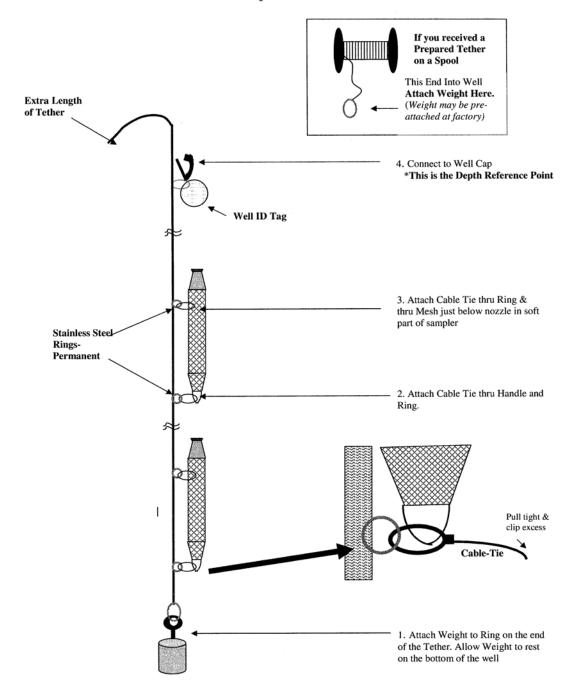
BASIC USE INSTRUCTIONS* (Fig 1)

- Fill the Sampler with deionized water until the entire assembly is completely full of water. To use the funnel, insert the tip into the Sampler and pour deionized water into the tube. Fill the Sampler until water rises and stands at least two inches up the funnel to expand the Sampler to its maximum capacity. Gently squeeze and add more water to expand the membrane and remove air pockets. Repeat as needed until completely full. Disclosure Statement - When filling the Sampler, we recommend that you hold the Sampler firmly at the top as close to nozzle tip as possible to prevent unnecessary stress on inside poly bag which could cause a leak to
- Insert the Plug firmly into the Sampler, until the rim of the plug is as close to the nozzle as possible.
- Attach a Weight to the bottom of the Tether or Hanger.
- Attach the Equilibrator(s) to the Tether line. If installing on a factory prepared tether, locate the small (1/2" diameter) stainless steel rings that are attached to the Tether line. The rings will be separated by approximately 2/3 the length of the sampler. Use a Cable-Tie through the lower of two adjacent rings and through handle. Use a second Cable-Tie through upper of two adjacent rings and through a section of mesh below the fill nozzle in the softer part of the filled sampler. Tighten the Cable-Ties and snip off excess. Continue with each Sampler. If the factory did not prepare the Tether, then securely attach the Sampler(s) to the tether using cable ties at the
- Lower the Tether with Sampler(s) attached into the well. Locate Sampler(s) below the water surface, in the screen flow zone of the well. Attach the top of the suspension cord to a well cap or other secure location at the top of the well. Leave Sampler in place for a time suitable for equilibration, a minimum of 2 weeks required.
- Upon retrieval: Discharge sample immediately to avoid loss of volatile compounds. Select a point on the Sampler near the handle/bottom of sampler. Press one end of the Discharge Tube firmly into the clear polyethylene membrane at a downward angle until it pierces the membrane. Discharge small amount to waste to purge discharge tube.
- *Contact EON for detailed installation information and for factory prepared Tethers.

800-474-2490



EQUILIBRATOR TM Diffusion Sampler Instructions

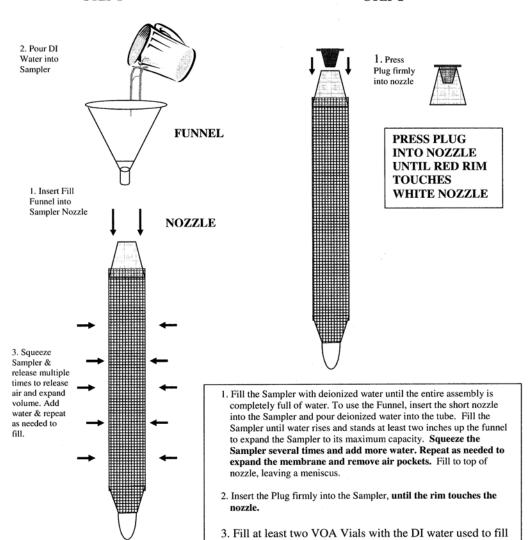




EQUILIBRATOR TM Diffusion Sampler Instructions

STEP 1

STEP 2



the samplers to use as a water blank. (Not Shown)

EPA SOP 2420.4C – Field Chain of Custody for Environmental Samples, December 2, 2003

STANDARD OPERATING PROCEDURE

No. 2420.4C

FIELD CHAIN OF CUSTODY FOR ENVIRONMENTAL SAMPLES

December 2, 2003

by Nicole Roblez

ENSV/RLAB/CATS

APPROVED: Peer Reviewer Peer Reviewer	13/3/43 Date
Manager, Regional Laboratory	<u>4 Dac 03</u> Date
Harold D. Brown Independent QA Reviewer	12/08/03 Date
Recertified	
Reviewer	
Date	

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- 1. RLAB Custody Seal; Total number of pages: 1.
- 2. Chain of Custody Record (COC); Total number of pages: 1.
- 3. Instructions for Completing a Chain of Custody Record (COC); Total number of pages: 3.

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A. Purpose

The purpose of this Standard Operating Procedure (SOP) is to establish uniform policies and procedures for use by field personnel to maintain an accurate written record of environmental samples from the time of collection through their acceptance by a laboratory for analysis. The custody procedures utilized within the laboratory for receiving samples and maintaining custody through the analytical processes are <u>not</u> covered in this SOP. See "Storage and Security of Environmental Samples", SOP 2420.2 for custody procedures utilized within the Regional Laboratory (RLAB).

B. Applicability

The policies and procedures outlined in this SOP are applicable to all Environmental Services Division (ENSV) personnel, Environmental Protection Agency (EPA), state/local agencies, and/or EPA contractors who collect environmental field samples for analyses by the RLAB or contract laboratories.

C. Summary of Procedures

As a requirement of any activity which may be used to support litigation proceedings, the validity of any data introduced into evidence must be clearly demonstrated. In the case of samples collected in support of an enforcement case, it must be clearly documented that the sample introduced into evidence is, in fact, the same sample collected and/or that the analytical data offered into evidence accurately represent the environmental conditions at the time of sample collection. It is imperative that there is adequate proof to demonstrate that transfer, storage or analysis, and that the analytical results were obtained from the same sample collected. Therefore, an accurate written record must be maintained to track the possession and handling Chain Of Custody Record (COC) (see Attachment 2) of each sample from the moment of collection through analysis and its introduction into evidence.

By definition, a sample is in "custody" if:

- 1. It is in one's actual physical possession; or
- 2. It is in one's view, after being in one's physical possession; or
- 3. It is locked up so no one can tamper with it, after being in one's physical possession; or
- 4. It is placed in a designated secured area

D. Definitions/Acronyms

ASR	Analytical Services Request
CLP	Contract Laboratory Program
COC	Chain of Custody Record

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ENSV	Environmental Services Division
EPA	Environmental Protection Agency

LIMS Laboratory Information Management System

PM Project Manager PO Project Officer QC Quality Control

RECAP Region 7 Environmental Collection and Analysis Program

ESAT Environmental Services Assistance Team

RLAB Regional Laboratory

RSCC Regional Sample Control Coordinator

SOP Standard Operating Procedure

SRN Sample Receipt Notice
Tags Sample container labels
UPS United Parcel Service

VOA Volatiles

E. Personnel Qualifications

Personnel performing this task should have a basic knowledge of the RLAB sample and records management procedures.

F. Responsibilities

1. Project Manager

- a. The Project Manager submits a completed Analytical Services Request (ASR) to the RLAB 30 days before initiation of the sampling activity.
- b. The Project Manager or designee (i.e., field contractor) ships and/or delivers properly collected, preserved, labeled, and packaged samples to the RLAB.
- c. The Project Manager or designee (i.e., field contractor) is responsible for the accuracy and completeness of all accompanying paperwork. If any changes are required as a result of the sampling (e.g., sample number changes, additional analyses, samples not collected, quality control (QC) code additions), the Project Manager or designee (i.e., field contractor) must see that these corrections are made on all paperwork.

All changes made to the paperwork (COC, sample tags, or field sheets) must also be made to the information contained in the LIMS. It is the responsibility of the Project Manager or designee to <u>supply</u> correct information so that the Regional Sample Control Coordinator (RSCC) can

SOP No. 2420.4C Page 5 of 8

properly process the samples into the LIMS. Whenever possible, any changes are made prior to the delivery of the samples. If necessary, the RSCC will assist the Project Manager when changes are noted prior to sample collection/delivery, concurrent to sample delivery or after.

d. The Project Manager must be available to help resolve any problems with the samples or must designate someone to do this for them in their absence. This requires that when delivering samples, the Project Manager or designee stays with the RSCC to answer any questions. Samples must not be just dropped off (unless after normal business hours).

The Project Manager or designee calls the RSCC close to the anticipated delivery date and/or time that samples are sent by courier (i.e., Federal Express) to confirm that samples have arrived and to answer any questions the RSCC may have.

2. RSCC

- a. The RSCC opens the ice chest (cooler) and utilizing the Infrared Digital Thermometer, checks the cooler temperature and records the temperature (in degrees Celsius) in the last row of the "Receiving Laboratory Remarks/Other Information" column on the COC (see Attachment 2).
- b. The RSCC verifies the presence of all samples, checks all documentation and signs the COC after all paperwork is complete and accurate.
- c. The RSCC works with the Project Manager to obtain correct information and puts the amended information into the LIMS.
- d. The RSCC notifies the Project Manager of problems which prevent acceptance of the samples by ENSV. RLAB maintains all samples received in a secure location including those pending reconciliation of problems.
- e. The RSCC logs samples into the LIMS and is responsible for the proper storage, tracking and/or distribution of the samples to the appropriate contract laboratories (this includes while the sample is in transit to the contract laboratory facility). The RSCC prepares an electronic Sample Receipt Notice (SRN) message for each activity received by the RLAB and routes it appropriately to the Environmental Services Assistance Team (ESAT), the Contract Laboratory Program (CLP) PO, or the Region 7 Environmental Collection and Analysis Program (RECAP) PO, CATS PM, ANOP PM, and appropriate back-up personnel.

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G. Procedures

1. In order to ensure adequate control and documentation of collected samples, the number of personnel handling the samples from the time of collection through delivery to RLAB should be limited.

- 2. The following actions must be accomplished in order to ensure that the relationship between the physical sample and the description of the sample is clearly, completely and accurately established, and that the custody of the sample is initiated from the time of actual sample collection.
 - a. A unique number is assigned to each sample (see "Identification, Documentation, and Tracking of Samples", SOP No. 2420.5) in order to relate the descriptive information to a physical sample. If a sample consists of several containers for analysis of different parameters from the same physical sample, the same number is used for each portion of the original sample.
 - b. A sample tag (sample container label) is securely attached to each container at the time of collection for specific instructions for filling out the sample tag (see "Identification, Documentation and Tracking of Samples", SOP No. 2420.5).
 - c. Custody of the sample is initiated at the time of collection by ensuring that the sample is in the sample collector's physical possession or view at all times, or is stored in a locked place where no one can tamper with it.

The sample collector is responsible for the collected samples until they are delivered to the RLAB.

- 3. Samples may be delivered to RLAB by the sampler or EPA contractor via courier or commercial carrier.
 - a. Sampler or EPA contractor-conveyed samples are those transported and delivered to RLAB. The coolers may be sealed or unsealed, but the sampler or EPA contractor must ensure that they are secured in the transport vehicle when he/she is not physically with the vehicle.
 - b. Samples may be delivered via courier (e.g., Greyhound). The cooler and sample containers must be transported with the lids secured. The transfer of possession of the samples must be recorded from the sampler or EPA contractor to RLAB.

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c. Samples may be shipped via commercial carrier (e.g., Federal Express, Airborne, United Parcel Service (UPS)) from the field to RLAB. The cooler and sample containers must be sealed at the time of shipment.

- 4. Samples are considered to be sealed when they are packaged in such a manner that would prohibit tampering or readily reveal any tampering, if it occurred.
 - a. A custody seal (see Attachment 1) may be used to secure the individual sample container, as appropriate to meet specific regulatory program requirements. These custody seals must be signed and dated by the sampler or EPA contractor when used to seal individual sample containers.
 - b. The use of a custody seal must be used to secure the openings of boxes, plastic bags, ice chests or coolers containing samples. These custody seals must be signed and dated by the sampler or EPA contractor when used to seal the shipping containers.
- 5. The COC (see Attachment 2) is initiated at the time of sample collection and must accompany all samples. The COC is utilized to document the transfer of a sample from the sampler or EPA contractor through receipt by the RSCC or designated back-up at RLAB.

RLAB instructions for the completion of the COC are outlined in Attachment 3.

- a. The transfer of possession of the samples would occur when the sampler or EPA contractor delivers the samples to RLAB, gives them to the courier who will deliver the samples to RLAB, or packs the samples in a sealed shipping container for shipment to RLAB via commercial carrier.
- b. The original and yellow copy of the COC will accompany the samples to RLAB. When the samples are conveyed by the sampler or EPA contractor, the COC may be hand carried. When the samples are delivered via courier or commercial carrier, the COC must be placed in a plastic document enclosure which is enclosed in the shipping container.
- 6. When samples are delivered to RLAB after duty hours, the samples and the COC will be placed in the refrigerator located on the back dock until acceptance by the RSCC or designated backup in accordance with the procedures outlined in "Storage and Security of Environmental Samples", SOP No. 2420.2.

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7. Once RLAB has accepted the samples, the responsibility for custody of the samples transfers to the RLAB personnel. Custody of the samples is maintained through analysis in accordance with the laboratory's internal control procedures.

- 8. The original of the completed COC is obtained by RLAB for inclusion with the permanent site activity files, and included with the final data transmittal sent to the Project Manager.
- 9. The yellow copy of the completed COC is returned to the Project Manager for inclusion in their appropriate activity files after all samples, for a given activity, have been accepted.
- 10. The custody seals or evidence tape associated with the specific samples or sample shipments are not retained.

H. Quality Assurance/Quality Control

A written tracking record (COC) is maintained from the time that the sample is collected to its transfer from the collection site to its laboratory destination. This record is used to demonstrate that sample possession has been secured and limited. Signed and dated custody seals placed over the access points of the sample shipment demonstrate that the contents of the samples have not been tampered with or compromised.

I. References

- US EPA, Region 7,"RLAB Procedures for Sample Receipt and Log-In", <u>Environmental Services Division Operations and Quality Assurance Manual</u>, SOP 2420.1
- US EPA, Region 7, "Identification, Documentation, and Tracking of Samples", <u>Environmental Services Division Operations and Quality Assurance Manual</u>, SOP 2420.5
- US EPA, Region 7, "Storage and Security of Environmental Samples", <u>Environmental Services Division Operations and Quality Assurance Manual</u>, SOP 2420.2

Attachment 1

RLAB Custody Seal

CUSTODY SEAL	Date	Signature
STORY THE STATES STATES	ENVIRON BANKLON	PAS GELING THE PROTECTION
ं Signature	AGENC Date	CUSTODY SEAL

Attachment 2

CHAIN OF CUSTODY RECORD ENVIRONMENTAL PROTECTION AGENCY REGION VII

CONTENTS OF SHIPMENT SAMPLE NUMBER TYPE OF CONTAINERS SAMPLED MEDIA TYPE OF CONTAINERS SAMPLED MEDIA TOTAL BOTTLE BO	
TYPE OF CONTAINERS SAMPLED MEDIA RECEIVING LABORATORY	
NUMBERS OF CONTAINERS PER SAMPLE NUMBER S	
	\neg
	一
	\neg
	ᅱ
	\dashv
	ᅥ
	\dashv
	\dashv
	\dashv
	\dashv
<u> </u>	
 	
<u> </u>	
DESCRIPTION OF SHIPMENT MODE OF SHIPMENT	
PIECE(S) CONSISTING OF BOX(ES) COMMERCIAL CARRIER: COURIER	-
ICE CHEST(S): OTHERSAMPLER CONVEYED (SHIPPING DOCUMENT NUMBER)	
PERSONNEL CUSTODY RECORD	
RELINQUISHED BY (SAMPLER) DATE TIME RECEIVED BY REASON FOR CHANGE OF CUSTODY	,
SEALED UNSEALED SEALED UNSEALED REASON FOR CHANGE OF CUSTODY	
ALLINGOISTICS S.	
SEALED UNSEALED SEALED UNSEALED REASON FOR CHANGE OF CUSTODY	~
RELINQUISHED BY DATE TIME RECEIVED BY	
SEALED UNSEALED SEALED UNSEALED	

Attachment 3

Instructions For Completing A Chain Of Custody Record

(Note: Each numbered item explains what is to be entered into that particular block moving from left to right, top to bottom of the document.)

- 1. <u>Activity Leader</u>. Enter the first initial and last name of the EPA Project Manager.
- 2. <u>Name of Survey or Activity</u>. Enter the activity number and/or Analytical Services Request (ASR) number (e.g., ERN07/900) for which the samples were collected.
- 3. <u>Date of Collection</u>. Enter the day, month, and year the samples were collected.
- 4. <u>Sheet</u>. Enter 1 of 1 unless there are more than one total sheets describing the shipment. If multiple sheets, enter the consecutive number of each sheet of the total number of sheets (e.g., 1 of 3, 2 of 3, 3 of 3).
- 5. Contents of the Shipment.
 - a. Enter the specific sample numbers, number of sample type containers per sample number and sample media in the appropriate column
 - (1) The ASR number and the individual sample numbers composing the shipment are entered in the "Sample Number" column (e.g., 2222-2). If more than one sheet is required, continue on additional sheets. For shipments of a large group of samples, it would be more appropriate and efficient to complete a separate sheet for each shipping container.
 - (2) The types of containers for each sample number are entered in the columns provided. The size should be entered above the container type, as appropriate. For Volatiles, the "VOA Set" refers to two=40 ml vials contained in the cubitainer which are collected for volatile organics analyses. The container types are modified, as necessary or appropriate, to describe sample containers.
 - (3) The sampled media for each sample number will be indicated by placing an "X" in the appropriate column. If the sample media is not listed, the actual media sampled should be entered in the "Other" column (e.g., wipe, sludge, air, biota, fish, etc.).
 - (4) The "Receiving Laboratory Remarks/Other Information" is to be used by the RLAB to indicate any problems with the shipment or condition of the samples upon receipt; e.g., custody seal on sample container or shipping container broken, a sample container broken in transit, a sample lost due to leakage during shipment, etc. The temperature of the shipping coolers(s) are to be recorded in the lower area of this column. This column may also be used to record other sample numbers for cross-referencing purposes (e.g., external sample number).

Attachment 3 Page 1 of 3

- b. After entering all of the above information, the total contents of the shipment should be indicated by marking out any remaining lines in this section. This can be accomplished either by drawing a line across the next line after the last entry and entering "None to Follow" or "Activity/ASR Complete," or by drawing a line across the next blank line or diagonally across the remaining lines in the section and entering "None to Follow" or "Activity/ASR Complete."
- 6. <u>Description of Shipment</u>. Enter the total number of pieces (e.g., samples or sample containers) packed in the total number of shipping containers (e.g., ice chests, boxes or other, which comprise the total shipment)(e.g., 12 pieces in 2 ice chests or 24 pieces in 2 boxes).
- 7. Mode of Shipment. Indicate the mode by which the samples are shipped to the RLAB by placing an "X" in the appropriate line preceding the specific mode in this block. If the shipment is via commercial carrier, the name of the carrier and the shipping document number (e.g., airbill) should be entered in the appropriate lines provided. This information may be entered by the sample shipper (sampler or individual to whom the sampler relinquished the samples), or the shipment receiver (lab sample custodian), as appropriate.
- 8. <u>Personnel Custody Record</u>. This portion of the form provides the record of changes of custody of the shipment (sample or group of samples) from the sample collector to the laboratory. To provide an adequate written record, all of the blocks should be completed as described below.
 - a. The sample collector will sign the first "Relinquished By" block when the samples are presented to another individual or commercial carrier.
 - (1) An "X" should be entered in the appropriate block to indicate whether the shipment is sealed or unsealed with a piece of completed custody seal tape, the date and time when the samples are relinquished should be entered in the appropriate blocks, and the reason for change of custody (e.g., transport to lab, receipt by lab, etc.) should be entered in the appropriate block.
 - (2) If the sampler is presenting the samples to a commercial carrier for shipment, the name of the carrier should be entered in the next available "Received By" block. The signature of a representative of the carrier is not required.
 - b. Each individual who received the shipment of samples will sign the next available "Received By" block and enter an "X" in the appropriate block to indicate whether the samples were received sealed or unsealed with a piece of completed custody seal tape. If the samples were shipped via commercial carrier, the individual receiving the samples (e.g., sample custodian at the RLAB) should enter the date and time the samples were received and the reason for change of custody (e.g., receipt by the RLAB) in the appropriate blocks.

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c. Each successive individual who relinquishes custody of the samples will sign the next available "Relinquished By" block, enter an "X" in the appropriate block to indicate whether the sample shipment is sealed or unsealed with a piece of completed custody seal tape, enter the date and time when custody is relinquished and enter the reason for change of custody in the appropriate blocks.

Attachment 3 Page 3 of 3



STANDARD OPERATING PROCEDURE

SOP No.: 4.07

SOP Category: HTRW

Revision No.: 1

Date: December 2010

Field Logbook Use and Maintenance

1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to describe the methods for use and maintenance of field logbooks. This procedure outlines methods, lists examples for proper data entry into a field logbook, and provides the standardized HydroGeoLogic, Inc. (HGL) format.

2.0 SCOPE AND APPLICATIONS

This procedure provides guidance for routine field operations on environmental projects. Site-specific deviations from the methods presented herein must be approved by the assigned HGL project manager and the HGL project quality assurance/quality control officer. Consult the project-specific planning documents for other documentation requirements that apply to the project.

3.0 GENERAL REQUIREMENTS

All work will be performed in a manner that is consistent with Occupational Safety and Health Administration established standards and requirements. Refer to the site- or project-specific health and safety plan for relevant health and safety requirements.

Personnel who use this procedure must provide documented evidence to the program manager or project manager that they have been trained on the procedure. This documentation will be retained in the project file.

Any deviations from specified requirements will be justified to and authorized by the project manager and/or the relevant program manager and documented in the planning documents. Deviations from requirements will be sufficiently documented to re-create the modified process.

All field personnel who travel to a site to conduct work related to environmental projects are responsible for documenting field investigation activities in project field logbooks in a legible manner and maintaining field logbooks over the course of the project in accordance with this SOP. Daily logs will be kept during field activities by the HGL field team leader, or approved designee, to provide daily records of significant events, observations, and measurements taken in the field.

The project manager or an approved designee is responsible for checking the field logbooks and verifying that they have been completed in accordance with this SOP.

SOP No.: 4.07

SOP Category: HTRW

Revision No.: 0
Date: December 2010

4.0 PROCEDURE

4.1 INTRODUCTION

Field logbooks provide a means for recording observations and activities at a site. Field logbooks are intended to provide sufficient data and observation notes to enable participants to reconstruct events that occurred while performing field activities and to refresh the memory of field personnel when writing reports or giving testimony during legal proceedings. As such, all entries will be as factual, detailed, and as descriptive as possible so that a particular situation can be reconstructed without reliance on the collector's memory. Field logbooks are not intended to be used as the sole source of project or sampling information. A sufficient number of logbooks will be assigned to a project to ensure that each field team has a logbook at all times.

4.2 FIELD LOGBOOK IDENTIFICATION

Field logbooks shall be bound books with consecutively numbered pages. Logbooks will be permanently assigned to field personnel for the duration of a project, or sampling event. When not in use, the field logbooks are to be stored in site project files. If site activities stop for an extended period of time (2 weeks or more), field logbooks will be stored in the project files in the appropriate HGL office.

The cover of each logbook will contain the following information:

- Organization to which the book is assigned (HGL)
- Project number (if different than site number)
- Book number
- Site name

4.3 LOGBOOK ENTRY PROCEDURES

Every field team will have a logbook, and each field activity will be recorded in the logbook by a designated field team member to provide daily records of significant events, observations, and measurements during field operations. Beginning on the first blank page and extending through as many pages as necessary, the following list provides examples of useful and pertinent information that may be recorded (optional).

- Serial numbers and model numbers for equipment that will be used for the project duration
- Formulas, constants, and example calculations
- Useful telephone numbers

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• County, state, and site address

Entries into the logbook may contain a variety of information. At a minimum, logbook entries must include the following information at the beginning of each day:

- Date
- Site name, site location, and project number
- Start time
- Weather
- All field personnel and subcontractors present and directly involved
- Level of personal protective equipment being used on the site
- Equipment used and calibration procedures followed
- Any field calculations

In addition, information recorded in the field logbook during the day will include, but is not limited to, the following:

- Sample description including sample numbers, time, depth, volume, containers, preservative, and media sampled
- Information on field quality control samples (e.g., duplicates)
- Sample courier airbill numbers and associated chains-of-custody
- Observations about site and samples (odors, appearances, etc.)
- Information about any activities, extraneous to sampling activities, that may affect the integrity of the samples
- Any public involvement, visitors, or press interest, comments, or questions; as well as times present at site
- Equipment used on site including time and date of calibration along with calibration gas/fluid lot numbers and expiration dates
- Background levels of each instrument and possible background interferences
- Instrument readings for the borehole, cuttings, or samples in the breathing zone and from the specified depth of the borehole, etc.
- Field parameters (pH, specific conductivity, etc.)
- Unusual observances, irregularities, or problems noted on site or with instrumentation used

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• Maps or photographs acquired or taken at the sampling site, including photograph numbers and descriptions

- A description of the investigation-derived waste (IDW) generated, the quantity generated, and the manner of IDW storage employed
- A photograph log that lists subject and persons, distance to subject, person taking photograph, distance, direction, time, photograph number, and noteworthy items for each photograph
- Forms numbers and any information contained therein used during sampling (Note that a form does not take the place of the field logbook.)

All logbook entries will be made in indelible black or blue ink. No erasures are permitted. If an incorrect entry is made, the data will be crossed out with a single strike mark and initialed and dated by the originator. Entries will be organized into easily understandable tables if possible. A sample format is shown in Attachment 1.

All logbook pages will be initialed and dated at the top of each page. Times will be recorded next to each entry. No pages or spaces will be left blank. If the last entry for a day is not at the end of a page, a diagonal line will be drawn through the remaining space and the line will be initialed and dated.

Logbooks can become contaminated when used in the field. Every effort should be made by the field team to avoid contaminating the logbook. Logbooks can be kept in seal-top poly bags or temporary plastic covers may be used.

4.4 REVIEW

The assigned project leader or an approved designee will check field logbooks for completeness and accuracy on an appropriate site specific schedule determined by the project leader. Any discrepancies in these documents will be noted and returned to the originator for correction. The reviewer will acknowledge that these review comments have been incorporated by signing and dating the applicable reviewed documents.

5.0 ATTACHMENTS

Attachment 1 Example Field Logbook

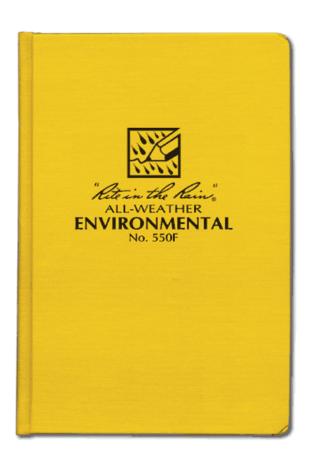
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ATTACHMENT 1 Example Field Logbook



ENVIR	ONMENTAL	4 x 4 to the inch with heading			
Lecellon Project / Client	Date	Project / Cliene	Dane		

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ATTACHMENT 1 (continued) Example Field Logbook

soluly 1800 mas	6,1995, A.	Madel # = 12345 Secret = 6789 FO Conductivity Metha	Model = 0345 Sande = 0789 (02 = 2 = 62 16 = 5	36.3	and longed home # 123-45107	T 4817 1	Directions to Site:	4 9 3
		PARORMATION RECORDED IN THE FRONT OF LOG OUNS (OPTIONAL) - stablishoods ** of equipment (present) - formula: contents, enumple rains - sate befores **	DAILY RECORDING REQUIREMENTS Intuits and date (up of every page) start time watcher decen methods (you may cross reference a previous days method if identical) personnel present on site	nativities recording into receives used spidons (time, depth. nations, preserv, etc.). (field and lab)		When using a field form information recorded a the field does not need to be written twice. Cross reference the field form # in the log book and record the information only on the appropriate field form.	DO MOT LEAVE ANY BLANK SPACESPAGES. If a page is sectionally left blank or there is united appear at the end of a 4475 entry days a diagonal line therugh the space and initial and dake the line.	

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ATTACHMENT 1 (continued) Example Field Logbook

N 11/45	33
November 6, 1995 Site Visit	- 1
Otov arrive on site	The samples will be taken from the
Weather: Bo, sunny, Slight breeze	ponds at the center of the dam
("5mph) from southwest.	opposite the outlets. (see below;
UDS Geld U Team: EPA 050:	refer to sample plans.
M. R. Smuth J. P. Scarten	All total Suspended solids (TSS) Samples
	will be collected in a 500 ml
P.R. Lane	polystyrene bottle - No preservative
PRP representative, L.M. Stein, Will	is necessary.
omogrania the USS Field	All VOA Samples unil be collected in
1	two 40-ml amber 9/855 vials and
be used	will be collected first. Preservation
	will be 4.0 (ice).
All equipment will be deconed as	- Meters (ett) Decon = Rinse with
Blows:	reagent-grade distilled water
- Brush equipment Brub brush to	
remove gross particulates.	PAND A
-	
water solution.	
- Rinse with reagent - grade distilled	
water.	The Day
- Rinse unith reagent-grade Methanol.	Outlet
- Phise with reagent-grade distilled	
Water.	location 35-1 @ Pord A.
Allow equipment to gravity deaven	0745: arrive @ POND A.
Wrap equipment in tinfail if not	Decon equipment as described
immediately used.	- 22
Sample procedure:	Calibrate of meter - Ringe probe
1.3	STO Reading
taken using a clean deconfaminated	7.00 7.00 1
TEFLON SCOOP; Stanless Steel gron	0754 4.00 4.00 Kinseprobe
and stainless steel wond un'll be	0754 Calibrate Conductivity Meter using
0-1	nomen - Dines probe

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ATTACHMENT 1 (continued) Example Field Logbook

4 10/45	10
Time Sample Sample Label #	FIELD PARAMETERS
	TIME PH Conductivity
TSS 8108 1354	10.00
	2 0
* laked 100 sell in mud-destroyed it.	of this logicook.
Field Parametrs	Fill out suctace water questity sheet.
10	,
10,35	0940 - Leave Pord 8 - head back
Deem commont (meters only)	to trailer to pack samples for
of Surface wa	Ship ment.
Le - wind speed !	0952 - armive at Trailer.
conds become	1
- leave Pond	forms for samples to be shipped
MA	WRAP Samples according to UOS
0840 - arrive at Pond B	
10	1030 - Seal Oboler and attach
,	3
	1030 - Take cooler to Federal Express
STO Reading	Por Shipping.
4.00 4.00	Cat # 1234567.
0845 7.00 7.00 Rinse Probe	1035 - Leave Pederal express.
0847 Calibrate conductivity meter	Sampling complete.
KSING 10000 STD - Ring probe.	
- 1	
Time Sample Sample # Label#	
	9
758	Charles and Charle
Ogos Decon scorp.	10
Time Sample Sample# Label#	
VOA 81088 VOAR	/

APPENDIX B

SAMPLE COLLECTION DATA SHEETS AND FIELD FORMS

- Daily Tailgate Safety Meeting
- Field Equipment Calibration And Maintenance Tasks
- Field Equipment Calibration And Maintenance Checklist (General)
- Field Equipment Calibration And Maintenance Checklist (Ysi)
- Field Equipment Calibration And Maintenance Checklist (Hanna)
- Observed Water Level And Well Integrity Inspection Form
- Passive Diffusion Bag Sampling And Deployment Form
- Pdb Field Parameter Form
- Field Sampling Report
- Accutest Laboratories Southeast Chain Of Custody

DAILY TAILGATE SAFETY MEETING

Meeting Conducted by:	Date and Time:	
Project Site:	Type of Work:	
Personal Protective Equipment:		
Chemical Hazards & Control Measures:		
Physical Hazards & Control Measures:		
Emergency Procedures:		
Hospital/Clinic:		
Address:		
Phone:		
	Attendees	
Name Printed	Signature	

Field Equipment Calibration and Maintenance Tasks

Instrument	Task	Frequency	Maintenance
	adjust meter to proper reading. Rinse electrodes and immerse in buffer solution (pH 4.0) and adjust meter to proper reading. Repeat the process until readings	pH will be checked with the 7 and 4 buffer solutions prior to the purging of the first well in each cluster.	
Cell water quality monitor unit or equivalent (see specific	Immerse the pH/ORP probe in the manufacturer's Zobell solution and follow the instruction to quality check or adjust the ORP reading Immerse conductivity sensor in calibration solution (provided by the manufacturer) and adjust calibration to the standard. Place the DO meter into the flow cell filled with approximately 1/8-inch of water. Allow to sit for approximately 10 minutes then follow the sequence of screens on the read-out box to calibrate. Calibration of the temperature sensor is not required per manufacturer's instructions.	Conductivity, ORP, and DO will be calibrated each morning prior to any sampling activities.	Check batteries; clean sediment from flow cell as needed.
Hanna HI98703 Turbidity Meter (see specific calibration form)	Calibrate the unit to the <0.1, 15, 100, 750 NTU AMCO or formazine standards.		Check Batteries
Water Level meter (general)	Check operation of the probe and circuits by turning on the water level indicator, inserting the probe into water, and listening for the indicator tone.		Daily Check Batteries

Field Equipment Calibration and Maintenance Checklist

Field Instrument	Calibration Readings	Pass?	Notes	Date	Initials

Field Equipment Calibration and Maintenance Checklist

EQUIPMENT MAINTENANCE AND CALIBRATION RECORD

Contract/Proje	ect:			Equipment Description	n: YSI 556	
Activity:				Equipment ID:	(Display	y Unit) & (Probe
				Equipment Serial No.:	(Disp	play Unit) & (Probe
Calibration Date/Time	Parameter	Standard Used (Concentration)	Lot Control No./ Expiration Date	Post Calibration Reading	Comments Pass/Fail	Signature
	PH	pH @ °C pH @ °C pH @ °C				
	ORP	Zorbell Solution mV @ °C				
	Conductivity	1409 <u>u</u> s/cm				
	DO	Air 100% Saturation				
	PH	рН @ °C рН @ °C рН @ °C				
	ORP	Zorbell Solution mV @ °C				
	Conductivity	1409 <u>u</u> s/cm				
	DO	Air 100% Saturation				
Notes/Mainter	nance Performed:					

Field Equipment Calibration and Maintenance Checklist

EQUIPMENT MAINTENANCE AND CALIBRATION RECORD

Contract/Project:			equipment Description:	·		
Activity.			Equipment Serial No.:			
Calibration Date/Time	Parameter	Standard Used (Concentration)	Lot Control No./ Expiration Date	Post Calibration Reading	Comments Pass/Fail	Signature
		0.1				
	Turbidity	15				
	Turbland	100				
		750				
		0.1				
	Turbidity	15				
	. a. D.a.i.y	100				
		750				
		0.1				
	Turbidity	15				
	. a. D.a.i.y	100				
		750				
Notes/Mainter	nance Performed:					

Observed Water Level and Well Integrity Inspection Form

Project: The Former St. Louis Ordnance Plant, Former Hanley Area, St. Louis, Missouri										
Project no:										
Personnel:										
						Well I	Inspection	$(\sqrt{,} *)$		
Well No.	Date	Time	Static Water Level (ft. btoc)	Total Depth of Well (ft. btoc)	Well Cap	Well Casing	Pad	Lock	Protect. Casing	Comments
							ļ			
		<u></u>								

Passive Diffusion Bag Sampling and Deployment Form

Project:	
Samplers:	

Well ID	Date Sampled	Time Sampled	Date Deployed	Time Deployed	Static Water Level (ft btoc)	Depth to top of PDB (ft btoc)	Water Column Over PDB (ft)	Observations
	1							
	1							
Analysis:			PDB Source: _			Water Source:		Pre-filled
			PDB Lot:			Water Lot:		Unfilled

PDB Field Parameter Form

ject:								
nplers:								
strument Used:								
		I	T		Water Quali	ty Parameters		
Well ID	Date	Time	Temperature	Specific Conductivity	Dissolved Oxygen	pH	ORP	Turbid
			(°C)	(µmhos/cm)	(mg/L)	su	(mV)	(NTU

FIELD SAMPLING REPORT

LOCATION: _			PROJECT:					
SITE:								
			INFORMATION	1				
MATRIX			SAMPLE ID: _					
SAMPLING M	ETHOD		DUP./REP. OF	:				
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			YES ()	N	0()			
END DEPTH _ GRAB ()		OSITE ()	DATE:		TIME:			
CONTAINER	2	PRESERVATIVE/	EXTRACTION	ANAL	YTICAL	ANAYLSIS		
SIZE/TYPE	X	PREPARATION	METHOD	MET	THOD			
			OBSERVATION	-				
PID READI	NGS	SAMPLE CHA	ARACTERISTICS MISCELLANEOUS					
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рНТ	Cemperatu	reDissolved	l oxygen	Speci	fic Condu	ctivity		
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WEATHED.		OLIVLIKAI	L IIVI OKWATIO	11				
WEATHER:		CLEAR OVERCAST/RA	AIN WIND DIR	ECTION _	AMBIE	ENT TEMP		
SHIPMENT VIA	FED-	X HAND DELIVER	R COURIER	C	THER			
SHIPPED TO: _								
COMMENTS: _								
SAMPLER:			OBSERVER:					
		PE CODES		PLING	METHO	O CODES		
DC=DRILL CUTTI WG=GROUND WA LH=HAZARDOUS WASTE SH=HAZARDOUS WASTE SE=SEDIMENT	TER LIQUID	SL=SLUDGE SO=SOIL GS=SOIL GAS WS=SURFACE WATER SW=SWAP/WIPE	B=BAILER BR=BRASS RING CS=COMPOSITE SAMPLE C=CONTINUOUS FLIGHT AUGER DT=DRIVEN TUBE W=SWAB/WIPE G=GRAB HA=HAND AUGER H=HOLLOW STEM AUGER HP=HYDRO PUNCH SS=SPLIT SPOON SP-SUBMERSIBLE PUMP					



Accutest Laboratories Southeast

Chain of Custody 4405 Vineland Road, Suite C-15 Orlando, Fl 32811

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DRAFT FINAL QUALITY ASSURANCE PROJECT PLAN THE FORMER ST. LOUIS ORDNANCE PLANT ST. LOUIS, MISSOURI

REGIONAL LTO/LTM FOR SEVEN INSTALLATIONS

Prepared for:



U.S. Army Corps of Engineers Kansas City District

Contract No. W912DQ-13-D-3000 Task Order 0004

Prepared by:

HydroGeoLogic, Inc. 6340 Glenwood Street, Suite 200 Building #7 Overland Park, KS 66202

January 2014

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LIST OF ATTACHMENTS

Attachment 1	Data Management and Validation
Attachment 2	HGL SOP 4.09 Data Validation
Attachment 3	Laboratory QA Manual

DRAFT FINAL QUALITY ASSURANCE PROJECT PLAN THE FORMER ST. LOUIS ORDNANCE PLANT ST. LOUIS, MISSOURI

REGIONAL LTO/LTM FOR SEVEN INSTALLATIONS

INTRODUCTION

This Quality Assurance Project Plan (QAPP) has been prepared to support long term operations (LTO) and long term monitoring (LTM) at the former St. Louis Ordnance Plant (SLOP), St. Louis, Missouri. It has been prepared in the Uniform Federal Policy for Quality Assurance Project Plans (UFP-QAPP) format. This document meets the requirements and elements set forth in the *DoD Quality Systems Manual for Environmental Laboratories*, Version 4.2 (QSM) prepared by the U.S. Department of Defense (DoD) in 2010, and the U.S. Environmental Protection Agency (EPA) guidance document entitled *Uniform Federal Policy for Quality Assurance Project Plans* (prepared by the Intergovernmental Data Quality Task Force in 2005). Note that the DoD QSM version 5.0 was released in July 2013; however, accreditation under version 4.2 is considered to be valid by the DoD until such time as current laboratory accreditation is due for renewal. It is anticipated that the project laboratory supporting the sampling performed at the former SLOP will be operating under accreditation obtained using the requirements of QSM version 4.2.

This QAPP and its attachments establish the procedures that will be implemented to ensure sampling and analytical activities related to the project definable features of work meet the project specifications and conform to the contract requirements and applicable regulations.

Section 3 of the Work Plan details the history, site description, and remedial activities for the former SLOP. Work Plan Figure 1.1 illustrates the site location and LTM monitoring locations.

QAPP Worksheets #1 and #2 Title and Approval Page

Draft Final Quality Assurance Project Plan (QAPP), The Former St. Louis Ordnance Plant, St. Louis, Missouri, Regional LTO/LTM for Seven Installations Document Title U.S. Army Corps of Engineers, Kansas City District (CENWK) Lead Organization Joseph Vilain, PhD, HGL Preparer's Name and Organizational Affiliation 6340 Glenwood, Building 7, Suite 200, Overland Park, Kansas, 66202; (913) 317-8860; jvilain@hgl.com Preparer's Address, Telephone Number, and Email Address January 2014 Preparation Date **CENWK Project Manager: Signature** Kale Horton / CENWK / January 2014 **Printed Name/Organization/Date** Lead Contractor's Project Manager: **Signature** Chris Williams / HGL / January 2014 **Printed Name/Organization/Date**

Site Name/Project Name: The Former St. Louis Ordnance Plant LTO/LTM

Site Location: St. Louis, Missouri

Site Number/Code: N/A Operable Unit: OU1 Contractor Name: HGL

Contract Number: W912DQ-13-D-3000

Contract Title: Regional LTO/LTM for Seven Installations

Work Assignment Number (optional): 0004

1. Identify guidance used to prepare the QAPP: UFP-QAPP; DoD QSM Version 4.2.

2. Identify regulatory program:

The Comprehensive Environmental Response, Compensation, and Liability Act, as amended by the Superfund Amendments and Reauthorization Act, *Management Guidance for the Defense Environmental Restoration Program,* (Office of the Deputy Undersecretary of Defense, 2001), the National Oil and Hazardous Substances Contingency Plan, the Resource Conservation and Recovery Act, the Defense Environmental Restoration Program, and Department of Defense and Army Policy.

- 3. Identify approval entities: <u>CENWK</u>
- 4. The QAPP is: programmatic or **project-specific**
- List dates of scoping sessions that were held:
 A kickoff meeting to discuss the scope of work (SOW) was held August 28, 2013.
- 6. List dates and titles of QAPP documents written for previous site work, if applicable: No former QAPP documents were available. Performance objectives are based on the *Final Long-Term Management/Land Use Control Implementation Plan Operable Unit 1, St. Louis Ordnance Plant, Former Hanley Area, St. Louis, Missouri,* prepared by Conti and CH2M Hill in September 2012 for CENWK, 88th Regional Support Command, and the U.S. Army Environmental Command.
- 7. List organizational partners (stakeholders): <u>Missouri Department of Natural Resources</u> (MDNR), U.S. Environmental Protection Agency Region 7 (EPA)
- 8. List data users: HGL; CENWK; EPA Region 7; MDNR; and the former SLOP

QAPP Worksheets #3 and #5 Project Organization and QAPP Distribution

Distribution:

The following is the distribution list for this QAPP.

QAPP Recipients	Title	Organization	Telephone Number	Email Address
Kale Horton	Project Manager (PM)	CENWK	816-389-3656	Kale.e.horton@usace.army.mil
Chris Williams	PM	HGL	913-317-8860	cwilliams@hgl.com
Justin Barker	CQCS	HGL	913-317-8860	jbarker@hgl.com
Jerrett Domling	Technical Lead,	HGL	913-317-8860	jdomling@hgl.com
Klaas Doeden	Technical Lead	HGL	913-317-8860	kdoeden@hgl.com
Jean Dent-Smith	Laboratory PM	Accutest Laboratories-Southeast	407-425-6700	jeans@accutest.com
Ken Rapuano	Project Chemist	HGL	703-736-4546	krapuano@hgl.com
Jeff Martin	Database Manager	HGL	703-736-4533	jmartin@hgl.com

The project organizational chart is presented in Figure 2.1 of the Regional Quality Control Plan submitted under separate cover.

QAPP Worksheets #4, #7, and #8 Project Personnel Qualifications and Sign-Off Sheet

ORGANIZATION: HGL

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature/Date
Chris Williams	Project Manager	B.S., Geology Experience: 26 years	P.G. See Project Management Plan (PMP), Appendix B Resumes	
Ken Rapuano	Project Chemist/DQCM	B.S., Chemistry M.S., Chemistry Experience: 25 years	CHMM See PMP, Appendix B Resumes	
Jerrett Domling	Technical Lead/SSHO	B.S. Environmental Science Experience: 16 years	CQMC See PMP, Appendix B Resumes	
Klaas Doeden	Technical Lead/SSHO	B.S. Geology M.S. Geological Engineering Experience: 20 Years	P.G. See PMP, Appendix B Resumes	
Jeff Martin	Database Manager	B.S., Chemistry Experience: 20 years	See PMP, Appendix B Resumes	
Justin Barker	Contractor Quality Control Supervisor	B.S Biology Experience: 20 years	CQMC See PMP, Appendix B Resumes	

B.S. - Bachelor of Science

U.S. Army Corps of Engineers, Kansas City District

CHMM - Certified Hazardous Materials Manager

CQMC - Contractor Quality Control Supervisor

DQCM - Data Quality Control Manager

M.S. - Master of Science

P.E. - Professional Engineer

P.G. - Professional Geologist

PMP - Project Management Plan

QAPP Worksheets #4, #7, and #8 (Continued) Project Personnel Qualifications and Sign-Off Sheet

ORGANIZATION: Accutest-Southeast

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature/Date
Jean Dent-Smith	PM	B.S., Marine Biology Experience: 30 years		
Svetlana Izosimova	QA/QC Officer	Ph.D., Colloid Chemistry Experience: 22 years		
Harry Behzadi	Laboratory Director	Ph.D., Chemistry Experience: 30 years		

B.S. - Bachelor of Science

P.E. - Professional Engineer

Ph.D. - Doctor of Philosophy

QAPP Worksheet #6 Communication Pathways

Communication Drivers	Responsible Affiliation	Name	Telephone Number	Procedure (Timing, Pathway to & from, etc.)			
Approval for project modifications; corrective actions; delay in sampling.	CENWK PM	Kale Horton	816-389-3656	All project documentation and related reports will be provided to the CENWK PM, either directly by the HGL PM or via the CENWK PM's designee(s). Project reporting is due to be completed within 35 days of completion of field work. Daily and weekly reports are due on the business day following the designation (next day for daily report, following Monday for weekly reports for work completed on the previous Friday). Email will be used if direct transmittal is not possible or cannot be performed in a timely manner.			
Receipt of analytical laboratory data	CENWK PM	Kale Horton	816-389-3656	Project analytical data and related reports will be reported to the CENWK PM by the HGL Project Chemist or designee within 30 days of receipt of the data. The CENWK PM will have approval authority over such reports and will communicate approval or comments to be addressed prior to approval to the HGL Project Chemist. Comments will be provided to HGL within two weeks of report receipt and responses will be submitted for approval within 7 days of receipt of comments. This ensures that data are complete for inclusion in the project reports due 35 days from completion of fieldwork. Data reports will be either transmitted electronically via email or via FedEx for hard-copies and reports on compact disk.			
Manage all project phases/field corrective actions related to well monitoring.	HGL PM	Chris Williams	913-317-8860	The HGL PM (or designee) will notify the client contact [PM or designee(s)] of field changes or modifications via phone, email, or fax by close of business on the next business day. Day-to-day field activities are the responsibility of the HGL PM and other HGL designees will provide daily updates to the PM via phone, fax, or email.			
Manage project- specific safety and health.	HGL Site Safety and Health Officer (SSHO)	Klaas Doeden	913-317-8860	Health and safety are the responsibility of all personnel. All personnel are trained to conduct project tasks as safely as practicable. Any issues will be communicated to the HGL SHSO for timely resolution. The HGL SSHO will ensure that day-to-day operations of the project are conducted safely. Verbal communication, when possible, will be used to ensure immediate action. Information may also be transmitted by phone or email.			

HGL-QAPP, The Former St. Louis Ordnance Plant, St. Louis, MO - Regional LTO/LTM

QAPP Worksheet #6 (Continued) Communication Pathways

Communication Drivers	Responsible Affiliation	Name	Telephone Number	Procedure (Timing, Pathway to & from, etc.)
Manage project quality control	HGL CQCS	Justin Barker	913-317-8860	Corrective actions needed for CQCS, as related to all project activities, will be communicated immediately to appropriate personnel via verbal, telephone, or electronic means. The HGL PM will be kept informed of related activities; however, the CQCS will not report to the PM to ensure no conflict of interest arises.
Manage analytical quality control	HGL Project Chemist	Ken Rapuano	703-736-4546	Corrective actions needed for field QC, as related to analytical sampling, will be communicated by the HGL Project Chemist to the HGL PM immediately upon identification of the issue so as to determine timely resolution. Satisfactory resolutions may require no further action. Laboratory QC is also overseen by the HGL Project Chemist. No data will be released until validation is complete and the HGL Data QC Manager has approved the release. If appropriate, the HGL Project Chemist may contact the CENWK Quality Assurance Officer for advisement.
Analytical QC and Field QC as related to Analytical QC	HGL Project Chemist	Ken Rapuano	703-736-4546	The HGL Project Chemist will consult with the laboratory to ensure that data received meet project needs. Additionally, the HGL Project Chemist will review daily field documentation, as related to analytical sampling, to ensure that documentation is accurate. The HGL Project Chemist will review and/or validate data, as appropriate, and write any required reports. Data reports will be submitted to the HGL PM for review and release to the client. Issues requiring consultation with the HGL QAM will be communicated to the HGL QAM within one business day of identification of the issue.
Laboratory QC issues.	Laboratory PM	See Worksheets #4, #7, and #8	See Worksheets #4, #7, and #8	QC issues with the samples will be communicated by the laboratory to the HGL Project Chemist within two business days of the occurrence. The HGL Project Chemist will determine if consultation with the HGL QAM is necessary to resolve any issues.

QAPP Worksheet #9 Project Scoping Session Participants Sheet

Name	Role
Dave Herwig, PMP, CHMM	HGL Program Manager (via telephone)
Chris Williams, P.G.	HGL Project Manager
Jerrett Domling, CQMC	HGL Technical Lead
Alan Rittgers, P.G.	HGL Project Support
Larry Braman	HGL Subcontracts Manager
Trudy Kearney	HGL Contracts Manager (via telephone)
Mark McGowan, CIH, CSP	HGL Corporate SHM
Dan Hearnen	USACE IRP Manager
Jonathan Harrington	AEC
Barry McFarland	88 th RSC
Jill Fraley	USACE Section Chief
Doug Mellema	USACE COR
Kale Horton	USACE TO 0004 Project Manager
Saqib Khan	USACE CMPSC Project Manager
Glenn Tisdale	USACE WSOW Project Manager
Brian Hughes	USACE Ft. Riley Project Manager
T.R. Shepherd	USACE Project Chemist
Brad Trost, P.E.	USACE Project Engineer
Andrew Gosnell	USACE Project Geologist
Cathy Forgét	USACE Safety and Health

Notes:

AEC - U.S. Army Environmental Command
CHMM - Certified Hazardous Materials Manager
CIH - Certified Industrial Hygienist
CMPSC - Charles Melvin Price Support Center
COR - Contracting Officer's Representative
CQMC - Construction Quality Management for
Contractors

CSP – Certified Safety Professional HGL – HydroGeoLogic, Inc. IRP - Installation Restoration Program

P.E. - Professional Engineer

P.G. - Professional Geologist

PMP - Project Management Professional

RSC - Regional Support Command

SHM - Safety and Health Manager

TO - Task Order

USACE - U.S. Army Corp of Engineers

WSOW - Weldon Spring Ordnance Works

QAPP Worksheet #10 - Problem Definition

Activities at the former SLOP will be conducted at OU1. A summary of the work scoped at the former SLOP is as follows:

- Conduct Project Management activities;
- Conduct quarterly sampling of 12 monitoring wells through 2014 and annual sampling from 2015 through 2018;
- Inspect and maintain the monitoring wells;
- Manage disposal of investigation-derived waste (IDW) purge water;
- Maintain the former SLOP data in the task order LTO/LTM project database; and
- Prepare quarterly and annual reports.

QAPP Worksheet #11.1 - Project Quality Objectives / Measurement Performance Criteria

The project objectives are listed in Worksheet #10. DQOs are used to define data quality requirements based on the intended use of the data. DQOs are qualitative and quantitative statements that:

- Clarify the treatment LTM objectives;
- Define the data necessary to evaluate LTM activities;
- Determine the appropriate method of data collection; and
- Specify the level of decision errors acceptable for establishing the quantity and quality of data needed to support the project decisions.

The overall QA objective for this project is to implement procedures for obtaining and evaluating data that meet the DQOs to ensure or confirm that the LTM criteria are accomplished. QA procedures are established to ensure field measurements, sampling methods, and analytical data provide information that is comparable and representative of actual field conditions, and that the data generated are technically defensible. Specifically, chemical data will be generated during the LTM activities to determine the quality of the groundwater and to determine whether the performance objectives set forth in the decision document for the former SLOP are achieved. Data that meet the QA objectives and goals will be deemed acceptable. Data that do not meet objectives and goals will be reviewed on a case-by-case basis to ascertain its usefulness. When possible, corrective actions will be taken to bring data within the QA acceptability goals.

QAPP Worksheet #11.2 - Project Quality Objectives / Measurement Performance Criteria

Project Quality Objective Methods and Analysis

Sample and analytical specifications must be appropriate to ensure that measurements can be quantified accurately at levels below the project action levels (PALs). The following worksheets outline the sample collection measures, analytical methods, and data quality elements required for this project:

- Worksheet #12: Method Measurement Performance Criteria Tables
- Worksheet #13: Secondary Data Criteria and Limitations Table
- Worksheet #15: Reference Limits and Evaluation Tables
- Worksheets #19 and #30: Sample Containers, Preservation, and Hold Times
- Worksheet #22: Field Equipment Calibration, Maintenance, Testing, and Inspection Tables
- Worksheet #23: Analytical SOP References Table
- Worksheet #24: Analytical Instrument Calibration Table
- Worksheet #25: Analytical Instrument and Equipment Maintenance, Testing, and Inspection
- Worksheets #26 and #27: Sample Handling, Custody, and Disposal
- Worksheet #28: Analytical Quality Control and Corrective Action
- Worksheet #34: Data Verification and Validation Inputs
- Worksheet #35: Data Verification Procedures
- Worksheet #36: Data Validation Procedures
- Worksheet #37: Data Usability Assessment

QAPP Worksheet #12 Measurement Performance Criteria Tables

12.0 MEASUREMENT PERFORMANCE CRITERIA

The overall QC objective for this project is to develop and implement procedures for sample collection, laboratory analysis, field measurement, and data reporting that will provide data of a degree of quality consistent with its intended use as described in the DQO process (Worksheet #11). Worksheet #12 and the associated method-specific table (Worksheet 12.1) present the performance criteria for the analytical measurements performed in support of this project.

12.1 DATA QUALITY INDICATORS

Measurement performance criteria usually are expressed in terms of the data quality indicators (DQIs) precision, accuracy, representativeness, completeness, comparability, and sensitivity, which are known collectively as PARCCS. Of the PARCCS parameters, precision, accuracy, completeness, and sensitivity can be quantitatively measured and assessed. The parameters of comparability and representativeness are primarily qualitative in nature.

12.1.1 Quantitative Data Quality Indicators

Quantitative DQIs can be measured and assessed by performing QC checks and evaluating the results against numerical acceptance criteria. Where available, the method- and matrix-specific measurement performance criteria that are presented in the QSM will be used by the off-site laboratories to control quantitative DQIs. Where the QSM does not list QC criteria, the control limits for routine analyses will be used by the project laboratory. These QC limits will be sufficient to ensure that the analytical methods are performed under acceptable conditions and that results can be used as reported for the intended purposes, as described in Worksheet #37.

12.1.1.1 Precision

Precision is the measure of variability between individual sample measurements under prescribed conditions. Precision can be assessed by replicate measurements of known laboratory standards and by analysis of duplicate environmental samples (spiked or unspiked). Precision is determined by evaluating the relative percent difference (RPD) between duplicate sample results. Replicate measurements of known standards (laboratory control sample [LCS]/laboratory control sample duplicate [LCSD] pairs), spiked samples (matrix spike [MS]/matrix spike duplicate [MSD] pairs), and laboratory duplicate analyses are routinely monitored by the laboratory by comparing the RPD with established control limits. The formula for calculating RPD is:

$$RPD = \frac{|S-D|}{\frac{(S+D)}{2}} x100$$

where:

S = first sample value (original sample, LCS, or MS value) and

D = second sample value (duplicate sample, LCSD, or MSD value).

12.1.1.2 Accuracy

Accuracy is the degree of agreement of a measurement to an accepted reference or true value. An evaluation of the accuracy of a measurement system provides an estimate of measurement bias. Overall analytical accuracy is assessed on a batch-specific basis by evaluating the percent recovery (%R) of known concentrations for each analyte in the LCS (and LCSD) against the QC limits. One known reference standard or LCS is analyzed for every batch (maximum of 20 samples). The accuracy of specific sample analyses is assessed by evaluating the %R of the surrogate spike compounds (organic analyses). The %R QC criteria for MS/MSDs will be used to assess the potential for matrix interferences. The formula for calculating %R is:

$$\%R = \frac{A - B}{C} \times 100$$

where:

A = the analyte concentration determined experimentally from the spiked sample,

B = the background level determined by a separate analysis of the unspiked sample (for calibration standards, LCSs, and surrogate compounds, the value of this term is zero), and

C = the amount of the spike added.

Accuracy is also measured using percent difference (%D) between a result and the expected value. %D is usually used to evaluate accuracy when the acceptance of a QC result is dependent on another analytical result and not on a pre-defined window of acceptance. The formula for calculating %D is:

$$\%D = \frac{A - B}{A} \times 100$$

where:

A = the original quantity measured and

B = the comparison quantity measured.

12.1.1.3 Completeness

Completeness is a measure of the amount of valid data obtained compared with the amount that was expected to be obtained under correct, normal conditions. It is calculated for the aggregation of data measured for any particular sampling event or other defined set of samples (such as by site). Valid data is data which is usable in the context of the project goals and DQOs. Completeness is calculated and reported for each method, matrix, and analyte

combination. The number of valid results divided by the number of possible individual analyte results, expressed as a percentage, determines the completeness of the data set.

Sampling completeness is defined as the percentage of analytical results obtained compared with the projected number of analytical results that would be obtained from all planned sample locations. Analytical completeness is defined as the percentage of valid (nonrejected) analytical results obtained from measurement systems compared with the total number of analytical results requested. The formula for calculating sampling completeness is:

Sampling Completeness = Number of Planned Data Points ×100% Number of Data Points Obtained

The formula for calculating sampling completeness is:

Analytical Completeness = Number of Acceptable Laboratory Measurements ×100% Number of Laboratory Measurements Reported

The overall completeness for each aspect of this project (as described in Worksheet #14) is defined as the sampling completeness multiplied by the laboratory completeness. Although the ideal of 100% data completeness may not be achieved for a dataset, that dataset may still be usable to make site-specific decisions. The impact of rejected or missing data on project decisions will be evaluated on a case-by-case basis in accordance with Worksheet #37 and Attachments 1 and 2. In addition to calculating overall completeness for project datasets, completeness can be evaluated as subsets of the overall dataset, including subsets selected by method, matrix, or analyte. The types of completeness evaluation performed for each project should be specified in the site-specific QAPP and should be selected based on DQOs.

Completeness is calculated at the end of the data validation process and generally is not used to evaluate ongoing data generation process. However, the potential impact on completeness is one of the deciding factors in determining the appropriate course of CA when sample results are affected by a QA discrepancy.

12.1.1.4 Sensitivity

Sensitivity is defined as the capability of a method or instrument to discriminate between measurement responses representing different levels of a variable of interest.

The chemical data generated for this project will follow the sensitivity limit conventions presented in the QSM, which include the detection limit (DL), the limit of detection (LOD), and the limit of quantitation (LOQ), which are defined on a matrix- and analyte-specific basis. The laboratory must perform quarterly confirmation of DLs, LODs, and LOQs; sensitivity limits that cannot be confirmed must be re-established at higher concentrations.

The QSM defines the DL as the smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration at the 99% level of confidence. At the DL, the false positive rate (Type I error) is 1%. DLs are specific to an individual determination performed at an individual laboratory.

The QSM defines the LOD as the smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative rate (Type II error) is 1%. In accordance with QSM conventions, nondetected analytical results will be reported as the numerical value of the LOD with the qualification "U."

The QSM defines the LOQ as the lowest concentration that produces a quantitative result within specified limits of precision and bias. The QSM requires each LOQ to be set at or above the concentration of the lowest initial calibration standard. Detected analytical results reported below the LOQ are qualified as estimated; detected analytical results at or above the LOQ can be used without qualification unless affected by a QC issue.

12.1.2 Qualitative Data Quality Indicators

The DQIs of representativeness and comparability have only a limited ability to be evaluated using QC analysis results. These DQIs are primarily controlled by project planning and execution.

12.1.2.1 Representativeness

Representativeness is the degree to which data accurately and precisely express a characteristic of a population, parameter variations at a sampling point, or an environmental condition. Although representativeness is a qualitative measurement, it is evaluated through a multi-step process beginning with evaluation of precision and accuracy data. Project design (see Worksheets #14 and #16) is one of the critical inputs that determine if the data collected are representative of the population sampled.

Representativeness of individual samples will be controlled by sample collection and handling in accordance with the requirements of Worksheets #14 and #16 and the HGL standard operating procedure (SOP) 4.09 Data Validation presented Attachment 2. The sample containers and preservation methods presented in Worksheets #19 and #30 will be used to ensure that samples arriving at the laboratory retain the appropriate degree of representativeness. The holding times presented in Worksheets #19 and #30 have been established to ensure that samples retain representativeness at the time of extraction and analysis.

Representativeness will also be assessed using field and laboratory blank samples. A method blank (MB) will be analyzed with every analytical or preparation batch (as appropriate to the analytical method) to determine potential contamination introduced during routine laboratory procedures. Trip blanks (TBs) and equipment blanks (EBs) will be collected to assess potential contamination due to field conditions. The assessment of blank samples will determine if compounds detected in the environmental samples are site-related or have been introduced through shipping, storage, field procedures, or laboratory procedures.

12.1.2.2 Comparability

Comparability expresses the confidence with which one data set can be compared to another. Comparability also involves a multi-step evaluation and can be related to accuracy and precision as these quantities are measures of data reliability. Data are comparable if site considerations, collection techniques, and measurement procedures, methods, and sensitivity limits are equivalent for the samples within a sample set.

The sample collection planned for this project is intended to extend an already-existing data set that has been collected over a period of years; consequently, comparability of analytical results from the planned sampling events with the results in the historical data set is of great importance. The analytical procedures used for this project will, in some cases, be updated versions of the procedures used in prior sampling event; however, these method updates are expected to have a minimal impact on data usability and the analytical results can be integrated with the existing data set.

12.2 DATA QUALITY CATEGORIES

The two general categories of data that will be generated for use in project decision-making are: (1) screening data and (2) definitive data. The data validation requirements for each matrix and analytical parameter and matrix are specific to each project data source and end use and should be described in the format presented in Worksheet #36. The screening and definitive data validation protocols for this project are presented in Attachment 1. The data usability evaluation procedures are summarized in Worksheet #37 and presented in Attachments 1 and 2.

12.2.1 Screening Data

Screening data are generated by rapid methods of analysis with less rigorous sample preparation, calibration, or QC requirements than are necessary to produce definitive data. Sample preparation steps may be restricted to simple procedures such as dilution with a solvent, instead of elaborate extraction/digestion and cleanup. Screening data may provide analyte identification and quantitation, although the quantitation may be relatively imprecise. Screening data may be considered of unknown quality without corresponding definitive confirmation data. Several screening methods identified for use in this project have no corresponding definitive method and results from these methods will not require confirmation.

Some methods that routinely produce definitive data can also produce screening level data if the data validation process is not performed or is reduced. This reduced level of data quality will depend on the end use of the data and this determination must be made on a site-specific basis. The analytical methods that will only be required to produce screening level data and the associated sample matrices are indicated in Worksheet #23 and Worksheet #36.

12.2.2 Definitive Data

Definitive data are generated using rigorous analytical methods, such as approved EPA reference methods. The data can be generated in a mobile or fixed-base laboratory. Definitive data are analyte-specific, and both identification and quantitation are confirmed. Definitive analytical methods have standardized QC and documentation requirements and produce data for which analytical error (bias) can be determined. In order for data to be classified as definitive, the data must be validated after the results are reported in order to verify that the appropriate QC measures were taken and were in control. Also, the sample must be collected in a manner that is representative of current site conditions. Sample collection in accordance with the procedures presented in Worksheets #14 and #16 and the applicable SOPs provided in the Field Sampling Plan (FSP) is required for the associated results to be considered definitive; any discrepancies will require review and evaluation of the impact on final data use. Definitive data are not restricted in their use unless quality problems identified in the validation process require data qualification. The analytical methods that will be required to produce definitive level data are indicated in Worksheet #23 and Worksheet #36.

12.3 MEASUREMENT PERFORMANCE CRITERIA TABLES

The data quality elements presented in the Worksheet #12 tables are divided into two broad categories: screening level elements and definitive level elements. Each data quality element is associated with one or more of the DQIs discussed in Section 12.1. In addition to the PARCCS parameters, some methods also include analyte identification as a DQI. Analyte identification is an essential performance component of those methods and is included even though is not a PARCCS parameter.

The analytical acceptance criteria presented in Worksheet #12 tables are linked to the data validation protocols presented in Attachment 1. Each project laboratory is required to ensure compliance with method and SOP requirements regardless of the level of data validation that will be performed on the resulting data. If a QC element does not meet control criteria, the appropriate qualifier, as defined in Attachment 1, will be applied to all associated results. The overall impact of QC discrepancies, including data gaps resulting from rejected data points, will be assessed in accordance with Worksheet #37 and Attachment 2.

12.3.1 Blank Evaluation

It should be noted that the Worksheet #12 tables present acceptance criteria for reporting data associated with low levels of blank contamination. It is acceptable for the laboratory to report analytical data with low levels of blank contamination meeting the Worksheet #12 acceptance criteria. However, during the data validation process, *all* detected values in blanks will be used to evaluate the associated sample data, *regardless of whether the reported blank results met the acceptance criteria presented in Worksheet #12*. This is the one of the few cases where QC data that meet *reporting* acceptance requirements may still result in qualification of the associated data.

12.3.2 SOP Reference Structure

Sampling, extraction, and analytical method SOPs are referenced in the Worksheet #12 tables using a numbering system in the format "[letter]-[number]". Field sampling SOPs are designated "S-[number]" and other field SOPs are designated "F-[number]"; the corresponding SOPs are identified in Worksheet #21 and presented in Appendix A of the FSP. Laboratory SOPs are designated "L-[number]" for analytical methods and are qualitatively identified in Worksheet #23. All SOPs identified in Worksheet #23 are included in the laboratory QAP, which can be provided upon request.

QAPP Worksheet #12.1 Measurement Performance Criteria Table – Volatile Organic Compounds (VOCs) by SW-846 Method 8260B

Analytical Group	Gas chromatography/mass spectrometr	ry (GC/MS)	-	
Analytical Method/SOP ¹	L-1			
Matrix	Aqueous			
Sampling Procedure ²	S-2			
Data Quality Indicator (DQI)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	Frequency of QC Check	QC Sample Assesses Errors for Sampling (S), Analytical (A), or Both (S&A)
Screening Level Data Qua				
Accuracy/Bias	Analyte-specific (see Worksheet #15.1)	LCS recoveries	1 per analytical batch (maximum of 20 samples)	A
		MS and MSD recoveries	1 per 20 field samples (selected by field team)	S&A
	Method-specific (see Worksheet #15.1)	Surrogate spikes	Every sample, blank, and standard (does not apply if dilution factor $\geq 5x$)	A
Precision	RPD ≤30%	MS/MSD RPD	1 per 20 field samples (selected by field team)	S&A
		LCS/LCS duplicate (LCSD) ³ RPD	1 per analytical batch (maximum of 20 samples)	A
	RPD ≤30%	Field duplicate analyses ⁴	1 per 10 field samples (selected by field team)	S&A
Accuracy/Bias and Representativeness	No analytes detected > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater)	Method blank (MB)	1 per analytical batch (maximum of 20 samples)	A
Accuracy/Bias and	No target compound concentrations	EB	1 per 20 field samples	S&A
Representativeness	>½ LOQ	ТВ	1 per cooler used to ship samples	S&A

QAPP Worksheet #12.1 (Continued) Measurement Performance Criteria Table – VOCs by SW-846 Method 8260B

Analytical Group	GC/MS			
Analytical Method/SOP ¹	L-1			
Matrix	Aqueous			
Sampling Procedure ²	S-2			
Data Quality Indicator (DQI)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	Frequency of QC Check	QC Sample Assesses Errors for Sampling (S), Analytical (A), or Both (S&A)
Sensitivity	DL for each analyte < LOD	DL study	Preliminary determination, confirmed quarterly	A
	LOD for each analyte below associated regulatory limits, preferably by a factor of ≥ 3	LOD study	Preliminary determination, confirmed quarterly	A
	$LOD \le LOQ$ for each analyte	LOQ study	Preliminary determination, confirmed quarterly	A
Completeness	≥95%	Data completeness check	After sampling and analysis complete	S&A
Definitive Level Data Qua	lity Elements			
Accuracy/Precision	For each analyte, percent relative standard deviation (%RSD) \leq 15% for mean relative response factor (RRF) or correlation ($\rm r^2$) \geq 0.990 for curve	Five-point calibration for all analytes (minimum of six points required if using r ² to evaluate)	Prior to sample analysis and recalibration as required	A
	%D ≤20% for each analyte	Second source calibration verification	1 per initial calibration	A
	%D ≤20% for each analyte	Continuing calibration verification (CCV)	Before each 12-hour analytical sequence, after instrument tuning	A
Sensitivity	LOQ for each analyte	At or below low concentration of calibration curve	Each initial calibration	A
Analyte Identification	Relative retention time (RRT) within ± 0.06 RRT units for each analyte and surrogate	Position set using the CCV	Once per initial calibration and at the beginning of the analytical shift	A
	Ion peaks within method-defined acceptance windows	Mass spectrometer tuning	Before each 12-hour analytical sequence	A
	Spectral match to reference spectrum	Mass spectrometer results	All positive results must be confirmed	A

QAPP Worksheet #12.1 (Continued) Measurement Performance Criteria Table – VOCs by SW-846 Method 8260B

Analytical Group	GC/MS											
Analytical Method/SOP ¹	L-1											
Matrix	Aqueous	Aqueous										
Sampling Procedure ²	S-2											
				QC Sample Assesses								
		QC Sample and/or Activity		Errors for Sampling								
Data Quality	Measurement Performance	Used to Assess		(S), Analytical (A),								
Indicator (DQI)	Criteria	Measurement Performance	Frequency of QC Check	or Both (S&A)								
Accuracy/Bias	Retention time within ± 30 seconds of retention time in the midpoint standard of the corresponding initial calibration	Internal standards	CCVs	A								
	Peak area within 50-200% of the peak area in the midpoint standard of the corresponding initial calibration	Internal standards	CCVs	A								
Accuracy/Bias	Retention time within ± 30 seconds of retention time in the corresponding CCV	Internal standards	Every blank, sample, and standard	A								
	Peak area within 50-200% of the peak area of the corresponding CCV	Internal standards	Every blank, sample, and standard	A								

¹ Reference number from QAPP Worksheet #23.

%RSD = percent relative standard deviation

CCV = continuing calibration verification DL = detection limit

DQI = data quality indicator

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GC/MS = Gas chromatography/mass spectrometry

LOD =-limit of detection

LOQ = limit of quantitation

MS = matrix spike

MSD = matrix spike duplicate

QC = quality control

VOC = volatile organic compound

² Reference number from QAPP Worksheet #21.

³ LCSDs are not a method requirement; however, if this information is provided, it will be evaluated.

⁴ For low-level results (detected value $\leq 5x$ LOQ) or when one result is a nondetection, the control limit is absolute difference \leq LOQ (water) or $\leq 2x$ LOQ (soil). Nondetected values will be assigned the nominal value of the LOD for making this comparison.

QAPP Worksheet #13 Secondary Data Criteria and Limitations Table

Secondary Data	Data Source	Data Generator(s)	How Data Will Be Used	Limitations on Data Use
Annual LTM Report	Conti and CH2MHILL, September 2012. Saint Louis Ordnance Plant Former Hanley Area	Ongoing LTM Monitoring	Determine trends in dataset	No restrictions for use as monitoring-level data (see Basewide QAPP, Section 1.4.2)
LTM and Land Use Control Implementation Plan – OU1	Conti and CH2MHILL, September 2012. Saint Louis Ordnance Plant Former Hanley Area	Identify contaminants of concern and associated screening values	Determine if contaminant exceedances exist	No restrictions for use as monitoring-level data (see Basewide QAPP, Section 1.4.2)

QAPP Worksheets #14 and #16 Project Tasks and Schedule

<u>Sampling Tasks</u>: Groundwater samples will be collected from sites OU-1. Worksheets #17 and #18 detail the specifics of the sampling tasks.

<u>Analysis Tasks</u>: All required sampling and analysis, including all required field QC samples, are included in Worksheet #18. All required analyses are listed in Worksheet #23.

QC Tasks: All matrices will have the following field QC samples collected and analyzed: duplicates, MS/MSDs, and EBs. TBs will be collected in association with sampling for VOCs. All analytical methods capable of providing definitive data will be controlled by initial and continuing calibration standards, tuning, surrogate spike results, LCSs, laboratory duplicates, and all other QC procedures defined in the project analytical methods and required to produce definitive data. Methods only capable of providing screening level data will be required to meet all associated QC criteria. The project laboratory is responsible for all complying with all applicable QC and instrument performance requirements regardless of the end use of the data or the level of data review that will be performed.

Secondary Data: The secondary source(s) are identified in Worksheet #13.

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<u>Data Management Tasks</u>: Analytical data will be uploaded to the project database after validation. The database will also include, but not be limited to field measurements, well construction details, well ownership information, and spatial data. The database will contain the former SLOP secondary data dating back to 2008.

Project Schedule / Timeline: The Project Schedule and Timeline are located in the Project Management Plan.

QAPP Worksheet #15 Reference Limits and Evaluation Tables

The following tables provide the analyte lists for the analytical methods that will be used for samples collected from the former SLOP. The associated limits for sensitivity and accuracy are also included in each method-specific table.

The accuracy control limits presented in the Worksheet #15 table are based on those presented in the DoD QSM for Environmental Laboratories, version 4.2 (DoD, 2010). For organic methods, HGL has adopted a convention of using a default minimum lower control limit (LCL) of 10 percent to establish a minimum non-zero standard of performance. Organic data will also use the default minimum upper control limit (UCL) of 120 percent (aqueous) or 125 percent (solid) established by the AFCEC QAPP version 4.0.02 (AFCEC, 2005). In those cases where the QSM lists an LCL or UCL below the default minimum, the default has been used. If the QSM marginal exceedance (ME) limit for an analyte is at or below the default LCL or UCL, no ME limit is allowed for that analyte at the affected end of the control limit range. Where the control limits are not specified in the QSM, the site-specific laboratory's internally derived control limits will be used. This is indicated in the method-specific Worksheet #15 tables with the designation "LAB." Laboratory-derived control limits and the ME limits calculated from them will also be subject to the default minimum LCL and UCL requirements.

In all cases, the laboratory is required to report concentrations at or greater than the DL as detected results. Results reported as detections with quantitation below the corresponding LOQ will be reported by the laboratory with the qualification of J to indicate that the result is considered an estimate due to being quantified below the calibrated range. Non-detected results and results below the corresponding DL will be reported by the laboratory as nondetected results quantitated as the LOD and qualified U. Laboratory-assigned qualifiers may be subsequently modified during the data validation process (see Worksheet #36 and Attachment 1).

The aqueous PALs presented in the following worksheets are the Final Target Groundwater Cleanup Goals determined by the CENWK, EPA, and the MDNR in the Decision Document for the site.

QAPP Worksheet #15.1 Reference Limits and Evaluation Table – VOCs in Water by 8260B

			ratory Se Limits (µg	•			Accuracy	_	Exceedance (R)
Analyte	CAS Number	DL	LOD	LOQ	MCL (μg/L)	RSL (µg/L)	Control Limits (%)	Lower Limit	Upper Limit
1,1,1,2-Tetrachloroethane	630-20-6	.23	.5	1		0.5	80-130	75	135
1,1,2,2-Tetrachloroethane	79-34-5	.24	.5	1		0.066	65-130	55	140
1,1,2-Trichloroethane	79-00-5	.2	.5	1	5.0	0.041	75-125	65	135
1,1-Dichloroethane	75-34-3	.3	.5	1		2.4	70-135	60	145
Benzene	71-43-2	0.21	0.5	1	5.0	0.39	80-120	75	130
Carbon tetrachloride	56-23-5	.31	.5	1	5.0	0.39	65-140	55	150
Chloroform	67-66-3	.26	.5	1	80(3)	0.19	65-135	50	150
cis-1,2-Dichloroethene	156-59-2	.24	.5	1	70	2.8	70-125	60	135
Methylene chloride	75-09-2	2	4	5	5.0	8.4	55-140	40	155
Naphthalene	91-20-3	1	2	5			70-130	60	140
Tetrachloroethene	127-18-4	.32	.5	1	5.0	3.5	45-150	25	165
trans-1,2-Dichloroethene	156-60-5	.23	.5	1	100	8.6	60-140	45	150
Trichloroethene	79-01-6	.31	.5	1	5.0	0.26	70-125	60	135
Vinyl chloride	75-01-4	.44	.5	1	2.0	0.015	50-145	35	165
Surrogates									
1,2-Dichloroethane-d4	17060-07-0	NA	NA	NA	NA	NA	70-120	NA	NA
4-Bromofluorobenzene	460-00-4	NA	NA	NA	NA	NA	75-120	NA	NA
Dibromofluoromethane	1868-53-7	NA	NA	NA	NA	NA	80-115	NA	NA
Toluene-d8	2037-26-5	NA	NA	NA	NA	NA	85-120	NA	NA

CAS = Chemical Abstracts Service

DL = detection limit

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LOD = limit of detection

LOQ = limit of quantitation

ME = marginal exceedance

NA = not applicable

PAL = project action level

%R = percent recovery

 $\mu g/L = micrograms per liter$

QAPP Worksheet #17 Sampling Design and Rationale

Groundwater Sampling

• Sample locations and sampling events are summarized in Table 2.1 of the FSP. Figure 2.1 of the FSP shows the well locations.

QAPP Worksheet #18 Sampling Locations and Methods/SOP Requirements Table

Sample locations are summarized in Table 2.1 and shown on Figures 2.1 of the FSP. Once a sufficient number of sampling rounds have been completed, the sampling regimen will be optimized, which may change the numbers of wells sampled, frequency of sampling, or the parameter suite.

QAPP Worksheets #19 and 30 Sample Containers, Preservation, and Hold Times

The following table includes all analytical methods that are listed in Worksheet #23. Prior to sampling at a site, the project laboratory will be provided with the list of tests to be performed and required turnaround times. The field sampling team should work with the project laboratory to identify samples for analytical methods that can be combined in the same sampling container to optimize sampling time and reduce shipping costs and sample waste.

Holding times expressed in hours should be evaluated based on time of collection to time of preparation or analysis, as measured in hours and minutes. For example, an unpreserved aqueous VOCs sample collected at 10:30 a.m. on October 8, 2013, must be analyzed or extracted no later than 10:30 a.m. on October 15, 2013. Holding times expressed in days should be evaluated on the basis of calendar days elapsed, with the sampling date considered day "0." For example, a soil PCB sample collected on October 8, 2013, must be extracted no later than October 22, 2013.

QAPP Worksheet #19/30.1 Sample Containers, Preservation, and Hold Times

Analytical Group	Concentration level	Sample Location/ID Number	Method	Sample Volume	Containers	Preservation Requirements	Maximum Holding Time (preparation/ analysis)
VOA	Low	See FSP Tables and Figure	SW846 5030B/8260B	5 mL	3-45mL VOA Vials w/teflon septum cap	<6°C; adjust pH <2 with HCl	14 Days- Preserved 7 days- Unpreserved

°C = degrees Celsius HCl = hydrochloric acid mL = milliliter VOA = volatile organic analysis

QAPP Worksheets #19/30.2 Project Laboratory Identification

Matrix	Analytical SOP	Data Package Turnaround Time	Laboratory/ Organization	Backup Laboratory/ Organization	Sample Delivery Method	Certifications Required
All	All	15 business days	Accutest Laboratories Southeast 4405 Vineland Road Orlando, FL 32811 407-425-6700 www.accutest.com	Not identified	Overnight shipping (air transport)	DoD ELAP MDNR

QAPP Worksheet #20 Field QC Summary

Field duplicate pairs will be collected at a rate of approximately 1 per 10 field samples; MS/MSD pairs will be collected at a rate of approximately 1 per 20 samples; and TBs will be collected at a rate of 1 per cooler shipped containing VOCs samples. EBs will be collected daily; however, if samples are collected from sampling ports, dedicated equipment, or equipment that will not be reused, EBs are not required. Ambient blanks are not planned; they will be collected at the discretion of the Field Team Lead (FTL) based on his or her judgment that the presence of potential VOCs contamination sources in the sampling area could contaminate samples.

The identification of field QC samples will be performed using the protocols discussed in Section 5.2 of the FSP.

Matrix	Analysis/ SOP Reference ⁽¹⁾	Field Samples ⁽²⁾	Field Duplicates	MS/MSDs	EBs	TBs	PT Samples	Total # Analyses
Aqueous	L-1 (8260B)	12	1 per 10	1 per 20	0	1 per cooler of VOCs	None Scheduled	16

- (1) Number references from the Project Analytical SOP References table (Worksheet #23)
- (2) All values approximate

EB - equipment blank

MS/MSD - matrix spike/matrix spike duplicate

PT - proficiency test

SOP – standard operating procedure

TB – trip blank

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QAPP Worksheet #21 Project Field SOP Reference Table

In order to ensure that all necessary SOPs are available for the field sampling team, copies of all SOPs listed in this worksheet are included in Appendix A of the FSP.

Reference Number	Title, Revision Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work?	Comments		
Field Sampli	ing SOPs Referenced in this QAPP						
S-1	SOP 2.01: Sampling Equipment Cleaning and Decontamination	HGL	Bailers, trowels, ISM tool, and bowls	No			
S-2	SOP 4.0 Passive Diffusion Bag Sampling	HGL	Passive Diffusion Bag	No			
Other Relevant Field Operation SOPs							
NA	SOP 4.07: Field Logbook Use and Maintenance	HGL	NA	No			

QAPP Worksheet #22 Field Equipment Calibration, Maintenance, Testing, and Inspection Table

Field Equipment	Calibration Activity/Status Check	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	Comments
Water level meter	Check operation of the probe and circuits by turning on the water level indicator, inserting the probe into water, and listening for the indicator tone.	Daily	Tone verified	repeat	Instrument operator	

QAPP Worksheet #23 Analytical SOP References Table

The following table provides a listing of the analytical methods that will be used for the site investigations associated with this QAPP. Accutest's analytical SOPs are proprietary business information and are not included in this QAPP. Accutest's SOPs can be made available for review on request.

Reference Number	Lab SOP Number	Title, Revision Number, and / or Date	Definitive or Screening Data	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
L-1	MS005	Analysis of Volatile Organics by EPA method 8260B, May 2012	Definitive	HP5890/5970, HP5890/5973, HP6890/5975	Accutest Laboratories Southeast, Inc., Orlando, FL	No
P-1	OP021	SOP for Sample Introduction via SW846-5030, Jun 2012	Preparation method	OI 4560/4552 Archon	Accutest Laboratories Southeast, Inc., Orlando, FL	No

QAPP Worksheet #24 Analytical Instrument Calibration Table

In all cases, the CA required in this worksheet will be the responsibility of the bench analysts and the laboratory Section Manager responsible for each method. Where an instrumental problem cannot be resolved by CA/routine maintenance, the affected instrument must be removed from service. After necessary repairs, the instrument will be recalibrated and determined to be fully functional before being cleared for return to service.

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria ¹	Corrective Action (CA)	SOP Reference ²
GC/MS	Five-point initial calibration for all target analytes (six points required for curve)	Initially; thereafter, as the continuing calibration fails	%RSD of calibration check compounds (CCCs) ≤30%; mean RRFs for each system performance check compound (SPCC) ≥ method-specific minima Target analyte evaluation: $r^2 \ge 0.990$ or %RSD ≤15% for each analyte	1) Evaluate system 2) Recalibrate as necessary All SPCC and CCC acceptance criteria must be met before accepting initial calibration; all SPCCs and CCCs must be included in the initial calibration even if they are not on a site-specific target list.	L-1 and L-2
	ICV (must be from a second source)	Following initial calibration	%R = 80 to 120%	 Evaluate system Recalibrate as necessary 	L-1 and L-2
	Instrument tuning	Every 12 hours; marks the beginning of an analytical sequence	Ion peaks meet method requirements	Halt analytical sequence Evaluate system Retune and recalibrate as necessary	L-1 and L-2

QAPP Worksheet #24 (Continued) Analytical Instrument Calibration Table

Instrument	Calibration	Frequency	Acceptance	Corrective	SOP
	Procedure	of Calibration	Criteria ¹	Action (CA)	Reference ²
GC/MS (continued)	CCV	Every 12 hours, after instrument tune	RRT within ±0.06 RRT units for each analyte and surrogate (it is acceptable to update RRT windows using the CCV to account for minor fluctuations or after routine instrument maintenance) CCCs %D ≤20%; RRFs for SPCCs ≥ method-specific minima Each target compound %D ≤20% Internal standard retention time within ±30 seconds and peak area within 50-200% of retention time and peak area in the midpoint standard of the corresponding initial calibration	1) Evaluate system 2) Clean system 3) Recalibrate if necessary 4) Reanalyze affected samples since the last in-control CCV All SPCC and CCC acceptance criteria must be met before accepting CCV; all SPCCs and CCCs must be included in each CCV even if they are not on a site-specific target list.	L-1

QAPP Worksheet #25 Analytical Instrument and Equipment Maintenance, Testing, and Inspection

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference ¹
HP5890/5970, HP5890/5973, HP6890/5975	Injector port, column maintenance, source cleaning	SW-846 8260B	Leak test, column and injector port inspection, source insulator integrity	Need for maintenance determined by passing calibration and BFB – see MS005	Passing BFB and CCV, passing Internal Standard response	Column clipping and/or reconditioning, seal and liners replacement, filaments and insulators as needed	Laboratory Analyst	L-1
¹ Worksheet #	23 presents the analy	tical SOPs.						

¹ Worksheet #23 presents the analytical SOPs.

QAPP Worksheets #26 and #27 Sample Handling, Custody, and Disposal

Sample shipment procedures will include overnight shipment by commercial courier or direct transport by commercial courier, laboratory courier, or field team. When samples are collected on a Friday, HGL will coordinate with the laboratory to ensure samples can be received at the laboratory on Saturday.

Sample Collection, Packaging, and Shipment

Sample Collection (Personnel/Organization): Site Staff/HGL

Sample Packaging (Personnel/Organization): Site Staff/HGL

Coordination of Shipment (Personnel/Organization): FTL/HGL; Sample Receipt Manager/Accutest

Type of Shipment/Carrier: See introductory text

Field Sample Storage (number of days from sample collection): Samples will be held in the field no longer than overnight unless prior arrangements have been made with the laboratory. Holding times must not be compromised by holding samples in the field.

Special Sample Shipment Considerations: See introductory text

Sample Receipt and Analysis (Applies to all Worksheet #23 SOPs)

Sample Receipt (Personnel/Organization): Sample Management Staff/Accutest

Sample Custody and Storage (Personnel/Organization): Sample Management Staff/Accutest

Sample Preparation (Personnel/Organization): Organic Preparation Staff, Inorganic Preparation Staff, and Bench Chemists/Accutest

Sample Determinative Analysis (Personnel/Organization): Bench Chemists/Accutest

Sample Archiving

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Sample Extract/Digestate Storage (number of days from extraction/digestion): For 60 days from data report release or as required on a site-specific basis

Biological Sample Storage (number of days from sample collection): As required on a site-specific basis

Sample Disposal

Personnel/Organization: Sample Management Staff/Accutest

Number of Days from Analysis: 60 from data report release; up to 6 months on sample-specific request from HGL

QAPP Worksheets #26 and #27 (continued) Sample Custody Requirements

Field Sample Custody Procedures (sample collection, packaging, shipment, and delivery to the laboratory):

HGL will maintain CoC records for all field and field QC samples. A sample is defined as being under a person's custody if any of the following conditions exist: (1) it is in his or her possession; (2) it is in his or her view after being in the individual's possession; (3) it was in his or her possession and is locked up; or (4) it is in a designated secure area after being in his or her possession.

Procedures to ensure the custody and integrity of the samples begin at the time of sampling and continue through transport, sample receipt, preparation, analyses, storage, data generation, reporting, and sample disposal. Records concerning the custody and condition of the samples are maintained in the field and laboratory records. All sample containers will be sealed in a manner that will prevent tampering or indicate tampering, should it occur. In no instance will sample containers be sealed with tape.

<u>Sample Labeling</u>: Each sample and well location will have a unique sample ID number assigned in accordance with the site-specific sample IDs presented FSP Section 5.2. Field QC samples will be identified in accordance with the ID protocols presented in Worksheet #20. The following information will be included on the label:

- Project ID
- Sample ID

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- Type of sample matrix
- Preservative added
- Date and time of collection
- Required analytical methods
- Sampler's initials

The samples labels will be placed on the sample containers so as not to obscure any QA/QC data on the bottles. Sample information will be printed in a legible manner using a permanent (indelible) ink marker or will be preprinted. Field ID must be sufficient to enable cross referencing with the appropriate sample documentation forms. CoC forms will be completed at the time of collection, including all required information and ensuring that the CoC information matches the information on the sample labels.

<u>Sample Packaging</u>: Preservation reagents will be added to sample containers before or immediately after collection of the sample, as indicated in Worksheets #19 and #30. The samples will immediately be placed on ice and will be kept chilled during the work day until packaged for shipment to the laboratory.

QAPP Worksheets #26 and #27 (continued) Sample Custody Requirements (continued)

Sample coolers will be supplied by the laboratory. When packaging samples for shipment, the cooler will be prepared by placing approximately 1 inch of vermiculite or other sorbent material in the bottom of the cooler. The cooler drainage plug will be closed and the cap will be sealed in place with duct tape. Sample containers will be placed inside sealed plastic bags as a precaution against cross-contamination caused by leakage or breakage. Bagged sample containers will be placed in the coolers in such a manner as to eliminate the chance of breakage during shipment. Ice in plastic bags will be placed in the coolers to keep the samples at 6 °C or less throughout shipment. Prior to sealing the cooler, the sampler's copy of the CoC forms will be detached and provided to the FTL for the project file. The remaining portion of the completed CoC forms will be attached to the underside of the cooler lid in a sealed plastic bag. The cooler will then be taped shut and at least two completed custody seals will be affixed across the gap between the lid and body of the cooler.

<u>Sample Shipment</u>: Samples collected in the field will be shipped to the laboratory as expeditiously as possible. Sample shipment will be performed in accordance with all applicable DOT regulations. The samples will be shipped to the laboratory by the procedures identified in this worksheet. Arrangements will be made between HGL and the contract laboratory point-of-contact for samples that are to be delivered to a laboratory on a weekend so that sample condition and holding times are not compromised. During transit, it is not always possible to control sample temperature; therefore, the laboratory will use a temperature gun during sample check in to measure the temperature of each sample.

Laboratory Sample Custody Procedures (receipt of samples, archiving, and disposal):

The designated sample custodian(s) and staff are responsible for samples received at the laboratory. In addition to receiving samples, the sample receipt staff is also responsible for documentation of sample receipt and storage before and after sample analysis. Summaries of the minimal laboratory receipt procedures are as follows:

- Upon receipt, sign, date, and document the time of sample receipt on the airbills or other shipping manifests received from the couriers.
- Sign the CoC form assuming custody of the samples. If a CoC form is not received with a set of samples, the laboratory will immediately notify the HGL PM.
- Inspect the sample cooler for integrity and then document the following information:
 - Type of courier and whether the samples were shipped or hand delivered (copies of the airbills are maintained).
 - Availability and condition of custody information.
 - Sample temperature.

- If the temperature of the samples upon receipt at the laboratory exceeds the temperature requirements, these exceedences will be documented in laboratory records, and the laboratory must contact the HGL PM immediately and document any decision regarding the potentially affected samples.
- Presence of leaking or broken containers and indication of sample preservation.
- Verify that the holding time has not been exceeded. If a sample has exceeded holding time, the HGL Project Chemist or PM must be notified.
- Match the sample container information (e.g., sample tag/label), CoC records, and all pertinent information associated with the sample. The sample custodian then verifies sample identity to ensure that all information is correct. Any inconsistencies are resolved with HGL through the Laboratory PM and CA measures are documented before sample analysis proceeds.

QAPP Worksheets #26 and #27 (continued) Sample Custody Requirements (continued)

Samples and extracts will be archived at the laboratory in accordance with this worksheet. The laboratory is also responsible for the proper management and disposal of all sample residuals and extracts, following all applicable federal, state, and local laws; rules; and regulations.

Sample ID Procedures:

All field samples will receive a unique sample ID designation as detailed in FSP Section 5.2. Sample IDs clearly differentiate field QC samples (including duplicates and MS/MSDs) and IDW samples from environmental samples.

CoC Procedures:

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Documentation of the CoC of the samples is necessary to demonstrate that the integrity of the samples has not been compromised between collection and delivery to the laboratory. A CoC record to document the transfer of custody from the field to the laboratory will accompany each sample cooler. All information requested in the CoC record will be completed. In addition, the air bill number assigned by the overnight courier will be listed on the CoC record or the general logbook. One copy of the CoC form will be retained by the samplers and placed in the project records file. The remaining pages will be sealed in a plastic bag and placed inside of the cooler. Upon receipt at the laboratory, the CoC forms will be completed and a cooler receipt form will be completed. It is the responsibility of the laboratory to document the condition of custody seals and sample integrity upon receipt.

The following sample-specific information concerning the sample will be documented on each CoC form:

- Unique sample ID number;
- Date and time of sample collection;
- Designation of MS/MSD;
- Preservative used;
- Analyses required;
- Name of collector(s);
- Serial numbers of custody seals and transportation cases, if used;
- Custody transfer signatures and dates and times of sample transfer from the field to transporters and to the laboratory or laboratories; and
- Bill of lading or transporter tracking number, if applicable.

In addition to the information above, the field team will record the source of sample (including name, location, and sample type) and any location-specific QC (such as field duplicates and ambient blanks) in the field logbook at the time of collection. Sample-specific information also will be recorded on sample-specific sample collection sheets and retained in the project file. Pertinent field data, such as groundwater stabilization parameters, will be recorded in the field logbook and on preprinted forms and retained in the project file.

QAPP Worksheet #28 Analytical Quality Control and Corrective Action

The following tables provide general guidance for the evaluation of QC analyses and the implementation of CA for out-of-control situations. The method-specific acceptance criteria are presented in the applicable table in Worksheet #12 and Worksheet #15.

QAPP Worksheet #28.1 Method QC Table – GC/MS, GC, and HPLC Methods

QC Element	Frequency	Method/SOP QC Acceptance Limits ¹	CA	Person(s) Responsible for CA	DQI
Screening Leve	l QC Elements				
MB	Every analytical batch (maximum of 20 samples)	Target analytes not detected > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater)	 Rerun Evaluate batch Reanalyze or qualify results as necessary 	Section Manager/ Laboratory Analyst	Accuracy/Bias and Representativeness
LCS (and LCSD, if performed)	Every analytical batch (maximum of 20 samples)	Analyte-specific %R and RPD acceptance criteria	 Rerun Evaluate batch Reanalyze or qualify results as necessary 	Section Manager/ Laboratory Analyst	Accuracy/Bias (and Precision)
MS/MSD	As indicated on CoC forms, and as required for batch control	Analyte-specific %R and RPD acceptance criteria (not applicable to air methods or if parent sample concentration ≥ 4x the spike level)	Evaluate MS/MSD to assess matrix interference Evaluate batch and qualify results as necessary	Section Manager/ Laboratory Analyst	Accuracy/Bias and Precision
Surrogate Recovery	Every sample	Surrogate-specific %R acceptance criteria	Rerun Reanalyze or qualify results as necessary	Section Manager/ Laboratory Analyst	Accuracy/Bias
Definitive Leve	l QC Elements – GC	/MS Methods			
Internal Standard Performance	Every sample	Peak area within 50-200% of the peak area in the corresponding CCV	Rerun Reanalyze or qualify results as necessary	Section Manager/ Laboratory Analyst	Accuracy/Bias
2 2 1 0 1 11 11 11 10 1		Retention time within ±30 seconds of the corresponding CCV			

QAPP Worksheet #28.1 (Continued) Method QC Table – GC/MS, GC, and HPLC Methods

QC Element	Frequency	Method/SOP QC Acceptance Limits ¹	CA	Person(s) Responsible for CA	DQI
RRT Position	Once per initial calibration and at the beginning of the analytical shift	RRT within ±0.06 RRT units for each analyte and surrogate reported in each sample and standard	 Correct problem Recalibrate instrument Reanalyze results as necessary 	Section Manager/ Laboratory Analyst	Analyte Identification
Mass spectrometer results	All positive results must be confirmed	Spectral match to reference spectrum	Analyst must evaluate results to confirm identification if spectral match does not meet criteria Section manager must review analyst's determination	Section Manager/ Laboratory Analyst	Analyte Identification

¹ Method-specific acceptance criteria are presented in the corresponding Worksheet #12 and #15.

QAPP Worksheet #29 Project Documents and Records

The following is a list of the kinds of site records that will be utilized and maintained for the project, as well as the personnel responsible for generating and verifying each record (see Worksheets #4, #7, and #8 for identities of HGL personnel). All records should be maintained in the HGL, laboratory, and other subcontractor (such as construction, design, or data validation firms) project files for a minimum of 5 years or longer as required by contract.

Record	Generation	Verification
Sample Collection Documents and Records	•	
Field notes (bound logbook)	Field staff	FTL
Sample documentation forms	Field staff	FTL
Tailgate safety meeting forms	SSHO	Corporate H&S Officer
Daily QC reports	FTL	PM
QC checklists	Field staff	FTL
CoC records	Field staff	FTL
Airbills	Field staff	FTL
Custody seals	PM	QA Officer
Corrective action forms	Field staff	PM
Photographs	Field staff	Database Manager
GIS data	GIS staff	GIS Manager
On-site Analysis Documents and Records		
Equipment calibration logs	Field Staff	FTL
Equipment maintenance, testing, and inspection logs	Field Staff	FTL
Equipment calibration logs	Field Staff	FTL
Field sampling data sheets	Field Staff	FTL
Waste disposal records	FTL	PM
Off-site Analysis Documents and Records		
Sample receipt, custody, and tracking records	Sample Receipt Staff	Laboratory PM
Standard traceability logs	Analytical Staff	Section Manager/QA Manager
Equipment calibration logs	Analytical Staff	Section Manager/QA Manager
Sample preparation logs	Analytical Staff	Section Manager/QA Manager
Analytical run logs	Analytical Staff	Section Manager/QA Manager
Equipment maintenance, testing, and inspection logs	Analytical Staff	Section Manager/QA Manager
Analytical discrepancy forms	Analytical Staff	Section Manager/QA Manager

QAPP Worksheet #29 (Continued) Project Documents and Records

Record	Generation	Verification			
Reported analytical results	Analytical Staff	Section Manager/QA Manager			
Reported results for standards, QC checks, and QC samples	Analytical Staff	Section Manager/QA Manager			
Data package completeness checklists	Analytical Staff/Section Manager	Laboratory PM/QA Manager			
Sample disposal records	Assigned Laboratory Staff	Laboratory Operations Manager/QA Manager			
Extraction and cleanup records	Analytical Staff	Section Manager/QA Manager			
Raw data (stored electronically)	Analytical Staff	Laboratory Database Manager/QA Manager			
Electronic database deliverables (EDDs)	Laboratory Database Manager	HGL Database Manager			
Telephone logs, emails, faxes, and correspondence	Laboratory PM	Laboratory Operations Manager			
Data Assessment Documents and Records					
Data validation reports	Data Validator	Data Validation PM/Project Chemist			
Automated data review reports	Data Validator	Data Validation PM/Project Chemist			
Database QC spreadsheets	Project Staff	Database Manager			
Data usability assessments	Project Chemist	PM			
Deliverables					
Project planning documents, including WP (QAPP, FSP,					
SPERP), PMP, APP, SSHPs, PIP,	PM	QA Officer			
Project deliverables, including monthly O&M Reports, QCSRs,					
Periodic Groundwater Monitoring Reports,	PM	QA Officer			
Telephone logs, emails, faxes, and correspondence	All project staff	PM			
Permits	All project staff	PM			
Site maps	FTL	PM			
Design documents	Graphics Staff	PM			
EDDs	Design Staff	PM			
	Project Database Staff	Database Manager			
Notes					

Notes:

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APP - Accident Prevention Plan

FSP - Field Sampling Plan

PIP – Public Involvement Plan

PM – project manager PMP – Project Management Plan

QAPP - Quality Assurance Project Plan

QCSR – Quality Control Summary Reports WP – Work Plan

QAPP Worksheets #31, #32, and #33 Assessments and Corrective Action

Assessments:

Assessment Type	Responsible Personnel and Organization	Number and Frequency	Estimated Dates	Assessment Deliverable	Deliverable Due Date
Review of QAPP, SOPs, and Site Safety and Health Plan with Field Staff	HGL FTL	Prior to sampling startup and with all new field staff prior to assignment	TBD	Completed acknowledgement signature pages	48 hours following assessment
Work performed in accordance with programmatic and site-specific QAPPs.	HGL FTL	Ongoing during all phases of fieldwork	TBD	Daily progress reports	24 hours following conclusion of business day
Logbook and Field Form Review	HGL FTL	Daily	TBD	NA; corrections will be made directly to reviewed documents	24 hours following assessment
Laboratory Assessment for Appropriate Certifications, Capacity, and QAPP Review with Staff	HGL Project Chemist	Prior to sampling mobilization, as new laboratories are contracted	TBD	Receipt of copies of certifications. Email traffic concerning lab capacity prior to sampling startup. QAPP sign-off sheet received from laboratory.	48 hours following assessment
Tailgate Safety Meeting	HGL FTL	Daily	TBD	Verbal debriefing and daily sign- off log. If a safety incident occurs, a Supervisor Injury Employee Report is completed.	Weekly; any safety incidents will be reported to the PM and Corporate H&S Officer immediately

QAPP Worksheets #31, #32, and #33 (Continued) Assessments and Corrective Action

Assessments (continued):

Assessment Type	Responsible Personnel and Organization	Number and Frequency	Estimated Dates	Assessment Deliverable	Deliverable Due Date
Field Sampling and CoC Form Review Against QAPP Requirements	HGL Sample Coordinator	Daily	TBD	Corrections will be made directly to reviewed documents; communication may be in the form of email	24 hours following assessment
Data Validation	HGL Project Chemist	Per SDG	TBD	Communication may be in the form of email traffic clarification of the analytical report or CAs due to deficiencies identified in the validation process.	24 hours following assessment
Laboratory Report Deliverables and Analytical Results Against QAPP Requirements	HGL Project Chemist	As discrepancies are identified in the validation process	TBD	Memorandum or email to PM and Project Chemist	72 hours following assessment

Assessment Response and Corrective Action:

_					
		Assessment			
	Individual(s)	Response	Time Frame for	Responsibility for	Responsibility for
Assessment Type	Notified of Findings	Documentation	Response	Implementing CA	Monitoring CA
Review of QAPP, SOPs, and	HGL FTL	Completed	48 hours following	HGL FTL	HGL FTL
Site Safety and Health Plan		acknowledgement	assessment		
with Field Staff		signature pages			
Work performed in	HGL PM	Interim CA	By close of same	HGL FTL	HGL PM and QA/QC
accordance with		documented pending	business day		Manager
programmatic and site-		final approval			
specific QAPPs					
Logbook and Field Form	HGL FTL	Corrections will be	NA	HGL FTL	HGL FTL
Review		made directly to			
		reviewed documents			

QAPP Worksheets #31, #32, and #33 (Continued) Assessments and Corrective Action

Assessment Response and Corrective Action (continued):

Assessment Response and		Assessment			
Assessment Type	Individual(s) Notified of Findings	Response Documentation	Time Frame for Response	Responsibility for Implementing CA	Responsibility for Monitoring CA
Laboratory Assessment for Appropriate Certifications, Capacity, and QAPP Review with Staff	HGL Project Chemist	Response to email or memorandum	48 hours after notification	Laboratory PM	HGL Project Chemist
Tailgate Safety Meeting	HGL FTL	Included as part of the process of the Supervisor Injury Employee Report	24 hours after notification	HGL PM	HGL Corporate H&S Manager
Field Sampling and CoC Form Review Against QAPP Requirements	HGL Sample Coordinator	Response to email	48 hours after notification	HGL FTL	HGL FTL
Data Validation	HGL Project Chemist	If required, laboratory reports will be amended and corrections noted in the analytical narrative and contained with the validation report.	1 business week	Data Validation PM	HGL Project Chemist
Laboratory Report Deliverables and Analytical Results Against QAPP Requirements	HGL Project Chemist	If required laboratory reports will be amended and corrections noted in the analytical narrative.	72 hours after notification	Laboratory PM	Laboratory QA Manager HGL Project Chemist

QAPP Worksheet #34 Data Verification and Validation Inputs

This worksheet lists the inputs that will be used during data verification and validation. Inputs include planning documents, field records, and laboratory records. Data verification is a check that all specified activities involved in collecting and analyzing samples have been completed and documented and that the necessary records (objective evidence) are available to proceed to data validation. Data validation is the evaluation of conformance to stated requirements, including those in the contract, methods, SOPs, and the QAPP.

			Validation					
		Verification	(conformance to					
Item	Description	(completeness)	specifications)					
	Planning Documents/Records							
1	Approved QAPP	X						
2	Contract	X						
4	Field SOPs	X						
5	Laboratory SOPs	X						
	Field Rec	ords						
6	Field logbooks	X	X					
7	Equipment calibration records	X	X					
8	CoC Forms	X	X					
9	Sampling diagrams/surveys	X	X					
10	Drilling logs	X	X					
11	Geophysics reports	X	X					
12	Relevant Correspondence	X	X					
13	Change orders/field work variances	X	X					
14	Field audit reports	X	X					
15	Field CA reports	X	X					
	Analytical Data	a Package						
16	Cover sheet (laboratory identifying information)	X	X					
17	Case narrative	X	X					
18	Internal laboratory CoC	X	X					
19	Sample receipt records	X	X					
20	Sample chronology (e.g., dates and times of	X	X					
	receipt, preparation, and analysis)							
21	Communication records	X	X					
22	Project-specific PT sample results	X	X					
23	LOD/LOQ establishment and verification	X	X					
24	Standards Traceability	X	X					
25	Instrument calibration records	X	X					
26	Definition of laboratory qualifiers	X	X					
27	Results reporting forms	X	X					
28	QC sample results	X	X					
29	CA reports	X	X					
30	Raw data	X	X					
31	Electronic data deliverable	X	X					

QAPP Worksheet #35 Data Verification Procedures

Verification Input	Description	Responsible for Verification
CoC (shipping)	CoC forms will be reviewed upon completion and verified against the packed sample coolers and site sampling requirements. This QC check will be verified by initialing the CoC form next to the shipper's signature. A copy of the CoC form will be retained in the project file and the original and one copy will be taped inside the cooler in a waterproof bag.	HGL FTL
Log review	Log reviews will be performed on a daily basis. This review will be performed to verify that all field monitoring equipment was maintained, calibrated, and operated properly. In addition, the review denotes all required information has been correctly documented in the field logbooks and sample documentation sheets.	HGL FTL
CoC (receipt)	CoC forms will be reviewed and compared to cooler contents. Any discrepancies (sample bottles, sample IDs, requested methods) will be communicated to the Laboratory PM for resolution with the HGL PM.	Laboratory Sample Receipt Manager Laboratory PM
Analytical data package	All data used to prepare analytical data packages will be reviewed at multiple levels throughout the laboratory. The requirements for this review process are described in the laboratory's quality manual. No data packages will be delivered to HGL without the necessary approval.	Laboratory QA Manager
Analytical data package	Ensure that the appropriate analytical samples have been collected, appropriate site identifications have been used, and the correct analytical methods have been applied.	HGL Sample Coordinator
Analytical data package ¹	Review the analytical reports to establish that all required forms, case narratives, samples, CoC forms, logbooks, and raw data have been included.	Data Validator (HGL or subcontractor)
ADR output and laboratory data reports (export)	All SEDDs will be verified against the import requirements of the project database prior to transmittal to HGL, as detailed in the Data Management Plan.	Laboratory Database Manager
ADR output and laboratory data reports (import)	The data validator will perform an evaluation of sample- and batch-related QC results (see Appendix B, Table B.1) for screening or screening and definitive QC elements, as required for each method on a site-specific basis. Review ADR output to ensure compliance with the validation protocols presented in Appendix B.	TBD, Staff Validator or subcontractor
Project database	All data qualifiers applied to the project database by manual entry will receive a 100% QC check for accuracy and completeness. Prior to final approval, each EDD output will receive a 10% QC check of electronically reported results against the hardcopy laboratory reports.	HGL Database Manager

¹ This verification step is performed as part of the data validation process described in Worksheet #36 and Attachment 1.

ADR = Automated Data Review program

CoC = chain of custody

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EDD = Electronic database deliverables

FTL = field team leader

PM = Project Manager

QC = quality control

SEDD = Staged Electronic Data Deliverable

QAPP Worksheet #36 Data Validation Procedures

Validation Stage	Matrix	Analytical SOP ¹	Validation Criteria	Data Validator
Data Review S	Step IIa			
Data Verification	All	All	Package Completeness Holding Times: Worksheet #19 Narrative: Additional items noted for resolution or clarification	TBD, Staff Validator, or subcontractor
Data Validation - Definitive	All	All	DQIs: Method-specific criteria presented in Worksheets #12, #15, #24, and #28 Qualification: QAPP Attachment 1, Table 1.a; screening and definitive level items	HSW (subcontractor)
Data Review S	Step IIb			
Senior Review	All	All	See Worksheet #37 and Attachments 1 and 2	HGL Project Chemist
Overall Assessment	All	All	See Worksheet #37 and Attachments 1 and 2	HGL PM

¹ Refer to Worksheet #23.

QAPP Worksheet #36 (Continued) Data Validation Procedures

An overview of the data validation process is presented in the following table. This process is described in full in Attachment 1.

Validation	T 7 1 1 1 1 1	D 14	D D 11.6 W11.4
Stage	Validation Input	Description	Person Responsible for Validation
Data Review Step	IIa		
Data Verification	Laboratory data reports (see Worksheet #35)	The validator will verify data package completeness, review case narratives, evaluate sample delivery and condition, and evaluate preparation and analysis holding times (Worksheet #19).	TBD, Staff Validator or subcontractor
Data Validation	Laboratory data reports	The data validator will perform an evaluation of sample- and batch-related QC results (see QAPP Attachment 1, Table 1.a) for screening or screening and definitive QC elements, as required for each method on a site-specific basis.	TBD, Staff Validator or subcontractor
Data Review Step	IIb		
Senior Review	Data validation reports	Senior review of reports to approve of all validation results and final qualifiers; overall evaluation of analytical performance against QAPP requirements.	HGL Project Chemist
Overall Assessment	Project documentation (Worksheet #33)	Complete project dataset and documentation: Determine whether the sampling plan was executed as specified (that is, the number, location, and type of field samples were collected and analyzed as specified in the Work Plan); evaluate whether sampling procedures were followed with respect to equipment and proper sampling support (for example, techniques, equipment, decontamination, volume, temperature, and preservatives).	HGL PM

QAPP Worksheet #37 Data Usability Assessment

Summarize the usability assessment process and all procedures, including interim steps and any statistics, equations, and computer algorithms that will be used: The data assessment team will perform the operations summarized in Worksheet #35 and Worksheet #36 to evaluate sampling team and laboratory compliance with the requirements with this QAPP. Evaluation activities will be documented in the QA reports listed in Worksheet #29 and will be used to assess the usability of project data in levels of detail ranging from an analyte- and sample-specific basis to the overall dataset for the sampling event. A full description of the activities listed in this summary is presented in Attachments 1 and 2. The DQIs and formulas used to evaluate data quality are presented in Worksheet #12.

Describe the evaluative procedures used to assess overall measurement error associated with the project: The assessment will include an evaluation of the QC elements relating to precision, accuracy, representativeness, comparability, completeness (both sample collection and analytical), and sensitivity (see Attachment 1). The impact of any data gaps resulting from sampling incompleteness or rejected data will be evaluated in a data quality evaluation included as an appendix to the project letter report.

Identify the personnel responsible for performing the usability assessment: HGL PMs, project chemists, and database managers.

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Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies: Evaluation activities will be documented in the QA reports listed in Worksheets #29. An overall assessment of the impact of data usability issues will be presented in the project report. The usability assessment will evaluate the overall dataset from each site.

ATTACHMENT 1 DATA MANAGEMENT AND VALIDATION

DATA MANAGEMENT AND VALIDATION

1.0 INTRODUCTION

After samples have been collected and analyzed, the data will be reviewed, reported, and validated. This appendix details the procedures that will be conducted to ensure that the data were collected and obtained in accordance with this Quality Assurance Project Plan (QAPP), applicable guidance documents, and good practices. The overall goal is to ensure that the data quality requirements of the project are met.

2.0 LABORATORY DATA MANAGEMENT REQUIREMENTS

Accutest Laboratories (Accutest) is responsible for providing complete and correct data to HydroGeoLogic, Inc. (HGL) for all requested analyses. The QAPP addresses the project-specific requirements for analyses in Worksheets #12, #15, #24, and #28. Following analysis of the samples, the laboratories will perform a series of steps to deliver an acceptable final data report to HGL.

2.1 DATA REDUCTION

Data reduction is the process for collecting and transforming measurements, through mathematical and/or statistical formulas, into final reportable measurements. The calculations may be performed manually or electronically. Data reduction is performed by the analyst and consists of calculating concentrations in samples from the raw data. The complexity of the data reduction depends on the analytical method and the number of discrete operations involved (e.g., extractions, dilutions, instrument readings, and concentrations). The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values. Calculations and data reduction steps for various methods are summarized in the respective laboratory standard operating procedures (SOPs) (see QAPP Attachment 3).

Copies of all raw data and the calculations used to generate the final results, such as bound laboratory notebooks, strip-charts, chromatograms, spreadsheets, and computer record files, are retained on file as specified in this QAPP. Should HGL determine that the laboratory's data reduction processes require an in-depth review, these calculations and the associated raw data will be provided to HGL on request.

2.2 DATA REVIEW

Data review is performed to assess whether quality control (QC) requirements were met. Project laboratories will perform data review on 100 percent of the data deliverables. No data may be released to HGL without the appropriate analyst and supervisory reviews being performed and documented.

The individual analyst continually reviews the quality of data by evaluating the results of calibration checks, QC samples, and performance evaluation samples. The analyst performs data review during, immediately following, and after the completed analysis. The laboratory supervisor, analyst, or data specialist performs a secondary review of the data. The data reviewer is trained by the quality assurance (QA) manager or section leader to perform the data review.

The analytical laboratory data reviewer who has the initial responsibility for the correctness and completeness of the data will conduct the first level of review, which may contain multiple sublevels of all project-related data. Data reduction, QA review, and reporting by the laboratory will be completed as follows:

- Raw data produced by the analyst will be processed and reviewed for attainment of QC criteria as outlined in the SOPs, laboratory QA manual, and established U.S. Environmental Protection Agency (EPA) methods, as well as for overall reasonableness. These general QC criteria will be modified by the requirements of this QAPP and the *DoD Quality Systems Manual for Environmental Laboratories* (QSM), version 4.2.
- After entry into the laboratory information management system (LIMS), a computerized report will be generated and sent to the laboratory data reviewer.
- The data reviewer will decide whether any sample reanalysis is required.
- Upon acceptance of the preliminary reports by the data reviewer, final reports will be generated.

The laboratory data reviewer will evaluate the quality of the work based on an established set of laboratory guidelines. This person will review the data package to ensure the following:

- Sample preparation information is correct and complete.
- Analysis information is correct and complete.
- The appropriate SOPs have been followed.
- Analytical results are correct and complete.
- QC samples are within project-specific control limits.
- Special sample preparation and analytical requirements have been met.

Documentation is complete when all anomalies in the preparation and analysis process have been documented.

The laboratory will perform the in-house analytical data reduction and QA review under the direction of the laboratory QA director. The laboratory program administrator (PA) is responsible for assessing data quality and advising the project manager of any data that were rated "preliminary" or "unacceptable," or other notations that would caution the data user of possible unreliability.

2.3 LABORATORY DOCUMENTATION

Analytical reports transmit final results, methods of analysis, levels of reporting, associated QC data, and method performance data. The laboratory will submit the data report for each sample delivery group using a reporting format that presents the information for a Stage 2B deliverable as described in the January 2009 *Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use* (EPA document 540-R-08-005). In addition, issues affecting the analytical process will be noted in the case narrative included in each report. The number of significant figures reported will be consistent with the limits of uncertainty inherent in the analytical method. Consequently, most analytical results will be reported to no more than two or three significant figures.

Data are normally reported in units commonly used for the analyses performed. Concentrations in liquids are expressed in terms of weight or activity per unit volume (e.g., micrograms per liter [$\mu g/L$] or milligrams per liter [m g/L]). Concentrations in solid or semisolid matrices are expressed in terms of weight or activity per unit weight of sample (e.g., micrograms per kilogram [$\mu g/kg$] or milligrams per kilogram [m g/kg]). Solid and semisolid matrices will also be reported on a dry weight basis. The sample-specific sensitivity limits (detection limits [DLs], limits of detection [LODs], and limits of quantitation [LOQs]) are reported adjusted for subsample size and percent moisture, as well as all appropriate concentration, dilution, and extraction factors.

If any analytical anomalies are encountered during the analyses (e.g., an out-of-control matrix duplicate), it will be documented in a case narrative, and copies of the sample discrepancy reports or corrective action reports must be included in the laboratory data reports.

2.4 LABORATORY RECORD-KEEPING

At a minimum, the laboratory will retain all data related to sample preparation and analysis, as well as general observations, in appropriate hardbound laboratory notebooks or files. Laboratory notebook pages must be reviewed, signed, and dated by the author and receive an independent secondary review by a peer or supervisor who signs/initials and dates the data pages.

Corrections to notebook entries are made by drawing a single line through the erroneous entry and writing the correct entry next to it. All corrections are initialed and dated by the individual performing the correction.

After delivering acceptable hard copy and/or electronic data deliverables, the laboratory will store the original project data for at least 5 years unless otherwise specified in the subcontract agreement.

2.5 LABORATORY ACCREDITATION

Project analytical data will be produced by Accutest Southeast, Orlando.

2.5.1 Department of Defense Requirements

This project requires that the analytical data be generated by a laboratory that has been accredited under the Department of Defense (DoD) Environmental Laboratory Accreditation Program (ELAP). This accreditation involves the successful completion of an on-site audit by an auditing firm contracted by the DoD and the evaluation of performance evaluation sample results. Accutest Laboratories Southeast is required to maintain current DoD ELAP accreditation for all analyses, matrices, and analytes applicable to this project throughout the duration of this work.

2.5.2 State Requirements

Missouri does not have an accreditation program in place for laboratories testing environmental samples, however Accutest Laboratories Southeast, maintains accreditation under the DoD ELAP program and the NELAP accreditation program.

2.5.3 Other Assessment and Audit Tasks

No subcontractor laboratory technical system audits are planned for this project; however, an audit may be performed at any time during this project at HGL's discretion or at client direction. In the event that laboratory performance does not meet QAPP requirements and/or significant data quality issues arise, HGL reserves the right to perform additional system/project audits at any time throughout the project.

3.0 SUBCONTRACTOR DATA MANAGEMENT REQUIREMENTS

Upon receipt of a laboratory data package and the associated electronic data deliverables (EDDs), HGL will perform data management tasks required to ensure that all analyses were performed in accordance with project requirements. The data management requirements include conducting data verification, data evaluation, and data validation to determine the usability of the data for the original project objectives. Data verification, data evaluation, and data validation are separate levels of review that can be performed by themselves or in conjunction with each other. Evaluation activities will be documented in the QA reports listed in Worksheet #33 and will be used to assess the usability of project data in levels of detail ranging from an analyte- and sample-specific basis to the overall dataset for the sampling event (see Attachment 2 of this QAPP).

3.1 DATA VERIFICATION

Initially, laboratory deliverables are received at HGL in both .pdf (laboratory data report) and EDD formats, as discussed previously. HGL will perform data verification on every report submitted by a laboratory. Upon receipt of the laboratory deliverables, a data management staff member will perform the following actions:

• The deliverable will be inspected to verify that results were received for each requested analysis for each sample. If a result is missing, the staff member will

determine whether the laboratory submitted a deficiency report that accounts for the missing data.

• The data deliverable will be inspected for completeness based on the requirements specified in this plan. Inspection will verify only that all required report elements are present, not that the data within the report are complete.

3.2 ELECTRONIC DATA MANAGEMENT

All analytical results must be submitted to HGL in a format that meets the requirements of a Staged Electronic Data Deliverable (SEDD) version 5.0, Stage 2a or later to support Stage 2A automated data review (ADR). Once HGL verifies that each EDD meets format requirements, it will be loaded into the project's electronic database management program as "unvalidated" for user access on the network. These analytical results will be considered preliminary until data validation is complete.

In all cases, the EDD will be preserved exactly as delivered as both a stored text file and as submission tables which are not altered. All files loaded will be available for download from the electronic library in an unaltered form, in order to ensure data integrity and traceability. Once HGL verifies that each EDD meets format requirements, it will be loaded into the project's Microsoft SQL Server database through a web portal. The database is hosted by and managed by HGL. This upload, which will be available only to authorized users, includes real time data screening. These analytical results will be considered preliminary until data validation is complete.

The EDDs will be compared to the pdf version of the laboratory data report by the HGL data management coordinator. HGL will perform this review on 10 percent of the electronic data results. If a discrepancy is identified, the laboratory will be required to correct the error.

3.3 DATA EVALUATION

Data evaluation is performed to assess whether the QC requirements for field duplicates, laboratory duplicates, equipment blanks, surrogates, matrix spikes (MSs)/matrix spike duplicates (MSDs), percent solids, method blanks, and laboratory control samples (LCSs) were met. Data evaluation will be performed on 100 percent of the laboratory deliverables generated during this program. This data evaluation procedure will be performed in conjunction with the data validation performed on each data report and is described below.

The first stage of data evaluation will be performed using the latest version of the ADR software program. The ADR software must be able to import a SEDD deliverable from the laboratory and export a SEDD deliverable (with the appropriate data review qualifiers). Following ADR, an AMEC chemist will evaluate ADR outputs and integrate those results into the manual review process in accordance with Appendix D of HGL's corporate data validation SOP.

3.4 DATA VALIDATION

Data validation is a systematic process to ensure that all chemical analytical information meets uniform requirements and to determine whether the usability and defensibility of the data are adequate for their intended use. Validation of analytical results will be performed using ADR by a chemist experienced in data validation, and another chemist will perform peer review of each data validation report. All applicable analytical data packages will be validated to ensure compliance with specified analytical methods, QA/QC requirements, data reduction procedures, data reporting requirements, and required accuracy, precision, and completeness criteria. Each validation report will be subject to peer review.

Data validation will be performed on 100 percent of the results for environmental samples. Validation will consist of a review of those elements that compose a Stage 2B data validation as described in January 2009 *Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use* (EPA document 540-R-08-005). The data will be validated against the acceptance criteria presented in Appendices F and G of the QSM, version 4.2, and other applicable guidance; a full list of the applicable data validation guidance is presented in Worksheet #36 of the QAPP.

Data validation guidelines are presented below in Table 1.a, and data qualifiers are defined in Table 1.b. The data validation guidelines are based on the requirements of the QSM, version 4.2, and the analytical methods. The qualification requirements and data qualifiers are based on the EPA National Functional Guidelines for data review. These guidelines were developed for the review of data generated using Contract Laboratory Program analytical methods, and the qualification guidelines presented in Table 1.a have been modified to accommodate differences between Contract Laboratory Program method requirements and the method requirements presented in the SW-846 methods and the programmatic QC requirements of the QSM.

Upon completion, the data validator will provide a data validation report and an annotated EDD that contains all final data result qualifiers. These data qualifiers will then be uploaded into the project database, which will then be made accessible to the HGL project team and will be available for upload.

Table 1.a
Data Qualification Guidelines

		Qualification of	Qualification of					
		Detected	Nondetected					
Control Parameter	Exceedance of Control Limits	Results ¹	Results ²	Associated Results				
Compliance of sample receipt condition checks and holding times								
	Evidence of frozen samples	J	UJ	All method results in affected sample or cooler				
Sample temperature	12°C ≥ temperature > 6°C	J	UJ	All method results in affected sample or cooler				
	temperature > 12°C	J	R	All method results in affected sample or cooler				
Sample headspace (Method L-1)	Bubbles ≥6 mm noted in sample containers if used for analysis	J	R	All results in affected sample				
Sample condition	Issues noted by field team, sample receipt department, or analyst	Validator judgment	Validator judgment	All method results in affected sample				
Holding time	Holding time exceeded	J	UJ	All method results in affected sample				
Holding time	Holding time exceeded by greater than a factor of 2	J K		All method results in affected sample				
Completeness and compliance checks of batch- and sample-related QC (screening level review)								
	Analyte concentration ≥ DL but below LOQ	F	Not applicable	Affected results in sample				
Analyte quantitation	Analyte concentration above calibrated range, no corresponding diluted result	J	Not applicable	Affected results in sample				
Method (preparation) blanks	Analyte detected ≥DL in blank: Multiply by 5 to obtain artifact threshold ³	Results below artifact threshold:	Not applicable	Affected analyte results in preparation batch				
Method (preparation) blanks: acetone, methylene chloride, and methyl ethyl ketone (Method L-1)	Analyte detected ≥DL in blank: Multiply by 10 to obtain artifact threshold ³	Results below artifact threshold:	Not applicable	Affected analyte results in preparation batch				
Trip blanks and ambient blanks (Method L-1)	Analyte detected ≥DL in blank: Multiply by 5 to obtain artifact threshold; not adjusted for sample- specific factors	Results below artifact threshold:	Not applicable	Trip blanks: Affected analyte results in all samples shipped in same cooler;				
Trip blanks and ambient blanks: acetone, methylene chloride, and methyl ethyl ketone (Method L-1)	Analyte detected ≥ DL in blank: Multiply by 10 to obtain artifact threshold; not adjusted for sample- specific factors	Results below artifact threshold:	Not applicable	Ambient blanks: Specific site association				

Table 1.a (continued) Data Qualification Guidelines

		Qualification of	Qualification of	
Control Decreased	E I CC II	Detected	Nondetected Results ²	A * . 4 . 1 D 14
Control Parameter	Exceedance of Control Limits	Results ¹	Kesuits ²	Associated Results
Equipment blanks	Analyte detected ≥ DL in blank: Multiply by 5 to obtain artifact threshold; not adjusted for sample- specific factors	Results below artifact threshold:	Not applicable	Affected analyte results in samples in the same sampling
Equipment blanks: acetone, methylene chloride, and methyl ethyl ketone (Method L-1)	Analyte detected ≥ DL in blank: Multiply by 10 to obtain artifact threshold; not adjusted for sample- specific factors	Results below artifact threshold:	Not applicable	event (same day)
Surrogate recovery (Methods L-1)	recovery > UCL LCL > recovery $\geq 10\%$ recovery < 10% 2 or more mixed high and low ($\geq 10\%$)	1 1 1	Not applicable UJ R UJ	All method results in affected sample ⁴
LCS recovery	recovery > UCL LCL > recovery ≥ ME recovery < ME	1 1 1	Not applicable UJ R	Affected analyte results in the preparation batch
LCS/LCSD RPD	RPD > CL	J	Not applicable	Affected analyte results in the preparation batch
MS/MSD ⁵ (Methods L-1)	recovery > UCL LCL > recovery Precision > CL	M M M	Not applicable UJ Not applicable	Parent sample only; evaluate applicability to other samples
Field duplicate RPD	Same as laboratory duplicate	Same as laboratory duplicate	Same as laboratory duplicate	Parent and duplicate samples only
Completeness and compliance chec	cks of instrument-related QC (definitive l	level review)		
Instrument tuning	Method tuning requirements	R	R	All sample analyses associated with affected tune
Initial calibration SPCC and CCC performance (Method L-1)	Method-specific criteria (Worksheet #24)	R	R	All analyte results associated with the initial calibration
Initial calibration linearity	Method-specific criteria (Worksheet #24)	J	UJ	Affected analyte results associated with the initial calibration
ICV (second source) performance	Method-specific criteria (Worksheet #24)	J	UJ	Affected analyte results associated with the initial calibration or analytical sequence

Table 1.a (continued) Data Qualification Guidelines

Control Donoundon		Qualification of Detected Results ¹	Qualification of Nondetected Results ²	Associated Deceller
Control Parameter	Exceedance of Control Limits	Results	Results-	Associated Results
CCV %D (Methods L-1)	Method-specific criteria (Worksheet #24)	J	UJ	Affected analyte results associated with the continuing calibration
CCV internal standards (Method L-1)	IS retention time outside ±30 seconds from corresponding CCV	R	R	Affected analyte results associated with the internal standard in the continuing calibration
Initial calibration SPCC and CCC performance (Method L-1)	Method-specific criteria (Worksheet #24)	R	R	All analyte results associated with the initial calibration ⁸
Internal standards (Method L-1)	IS area >200% or <50% corresponding area in associated CCV	J	UJ	Target analytes quantitated using affected IS in affected
	IS area <25% corresponding area in associated CCV	J	R	sample.
	IS retention time outside ±30 seconds from corresponding CCV	R	R	
Other validator actions				
Multiple results reported for an analyte in a single sample/method combination due to multiple dilution levels or reanalysis due to QC issue	NA; the validator will review the available data and associated QC results and determine the "best" data point for each analyte reported for each sample	Х	X	Applied to all results not selected as the "best" data point.
Calculation and transcription verification	Errors or inconstancies noted	Validator judgment	Validator judgment	Results associated with the error; also notify HGL or Weston Project Manager or Senior Chemist
General data review	QC element not performed	Validator judgment	Validator judgment	Results associated with missing QC element

Notes

U.S. Army Corps of Engineers, Kansas City District

- ⁽¹⁾ The priority of qualifiers for detected results is: X > R > U > J >no qualifier.
- ⁽²⁾ The priority of qualifiers for non-detected results is: X > R > UJ > U.
- (3) All project preparation and analytical methods have holding times greater than 72 hours and holding time compliance will be evaluated on the basis of elapsed calendar days.

%D - percent difference

%RSD - percent relative standard deviation

< - less than

> - greater than

 \leq - less than or equal to

CCB - continuing calibration blank

CCC – calibration check compound CL – control limit

DL - detection limit

ICB - initial calibration blank

ICV - initial calibration verification

IS - internal standard

LOD – limit of detection LOQ – limit of quantitation QC – quality control RPD – relative percent difference SDG – sample delivery group

SPCC – system performance check compound

UCL - upper control limit

⁽⁴⁾ MS/MSD and post-digestion spike results for an analyte are not considered applicable if the concentration in the parent sample is > 4x the spike concentration.

⁽⁵⁾ When comparing the results of a duplicate pair which consists of a detected result and a nondetected result, the numerical value of the non-detected result should be considered to be the LOD. Two results below the LOQ or a result below the LOQ and a nondetection are always considered to be in control.

Table 1.b Data Qualifier Definitions

Qualifier	Definition
	Confirmed identification. The analyte was positively identified at the reported concentration.
No qualifier	The reported concentration is within the calibrated range of the instrument and the result is
	not affected by any deficiencies in the associated QC criteria.
J	The analyte was detected at the reported concentration; the quantitation is an estimate.
D	The result is rejected due to serious deficiencies in the ability to analyze the sample and meet
R	QC criteria.
U	Not detected. The associated number indicates the analyte LOD.
UJ	Not detected. The associated number indicates the analyte LOD, which may be inaccurate.
v	Excluded. The data point is associated with reanalyses or diluted analyses and is excluded
X	because another result has been selected as the definitive result for the analyte.

ATTACHMENT 2 HGL SOP 4.09 DATA VALIDATION



STANDARD OPERATING PROCEDURE

SOP No.: 4.09

SOP Category: Data Quality

Revision No.: 00 Date: November 2012

Data Validation, Level II and Level III

1.0 PURPOSE

This standard operating procedure (SOP) provides information on the methodology and protocols required to perform review and validation of analytical data generated from the laboratory analysis of environmental media. This SOP is intended to provide general guidance for the evaluation of the quality control (QC) elements that are associated with analytical data. Project-specific criteria for data validation will be presented in the project's Quality Assurance Project Plan (QAPP), as will be the project-specific QC acceptance criteria. Users of this SOP are authors of QAPPs, preparers of electronic QAPPs (eQAPPs) supporting automated data review (ADR), data validators, and data users.

2.0 SCOPE AND APPLICATION

The terms "Level II" and "Level III" are applied to the two most common levels of data validation performed in support of HydroGeoLogic, Inc.'s (HGL's) environmental projects. Level II validation, which also can be termed "QC Review," consists of a review of sample receipt and condition, holding times, and sample-specific and batch-specific QC elements. Level III validation consists of all the elements of a Level II validation, with additional review of instrument and analytical system QC elements. Neither Level II nor Level III validation requires the review of raw data. In some cases, however, an individual laboratory's data report format will require the examination of raw data to provide information on a specific QC element that is more often found on a summary form.

The level of data validation to be performed on analytical results will be determined by HGL's project scope of work and will be presented in the project QAPP. Depending on the objectives for the project dataset, the actual validation performed on any given set of results will be determined on a sample- and analytical method-specific basis. Generally, Level III data validation will be performed on analytical results that are required to be considered definitive and usable for performing quantitative risk assessment, or which have the potential to be used in a future risk assessment. Level II data validation is performed to provide a general assessment of sampling and laboratory performance and does not result in data that that are usable for risk assessment. Level II validation is typically performed on data generated from long-term monitoring, operations and maintenance sampling, natural attenuation parameters, and compliance monitoring.

Level IV data validation involves a greater level of effort and builds on the Level III data validation procedures. Level IV validation involves recalculation of results, verifying transcription of raw data to summary forms, and examination of raw instrument results including standard

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¹ These levels were originally defined in an U.S. Environmental Protection Agency (EPA) document that has been withdrawn; however, the terms remain in common use in the environmental field.

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preparation logs, quantitation reports, chromatograms, and mass spectra. Level IV validation relies almost entirely on the validator's professional judgment and experience and is not covered by this SOP. No Level IV data validation tasks can be assigned to HGL personnel without the approval of an HGL senior chemist.

Data generated for waste characterization and data associated with QC samples generally require no validation unless anomalous results are noted. Federal, state, or program requirements may include performing a higher level of validation than is normally performed on any given sample or set of samples.

The QC elements that make up data validation Levels II and III are provided in Attachment A. The components of Level IV data validation are also provided for reference.

3.0 GENERAL REQUIREMENTS

3.1 PRE-REVIEW ITEMS

Prior to beginning validation of laboratory data reports, the data validator must obtain the following items and information from the project manager (or designee):

- 1. The correct billing code for data validation tasks;
- 2. The most recent version of all relevant QAPPs (including any basewide QAPP and QAPP addenda);
- 3. The level of data validation to be performed on the data (multiple levels are possible depending on end use of individual samples or the results from specific analytical methods);
- 4. The schedule and anticipated level of effort to complete validation tasks;
- 5. The identity of any field duplicate samples and the associated parent samples; and
- 6. The identity of any field blanks (equipment, trip, ambient, and material blanks) and the correct association protocol for each blank.

3.2 LABORATORY DATA REPORTS

The data reports produced by each laboratory will have substantial differences in presentation, structure, and formatting when compared to a data report produced by another laboratory, although some similarities will be present. The laboratory is required to provide data packages that will support the level of review that the associated data will undergo. Summary pages that provide all the validation level-specific information listed in Attachment A are preferred, although in some cases summary pages may need to be supplemented with information only available on instrument printouts or raw data.

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Before data validation, the validator should examine the laboratory data reports to ensure that all required information necessary to perform the required level of data validation is available and presented in a format that will support the validation effort. Familiarity with the laboratory's reporting conventions will improve the efficiency of the data validation process, and will also improve the quality of the validation, as the validator will be better able to identify QC discrepancies in the reported data and judge the effect on the associated sample results.

Control limits for surrogate recoveries, laboratory control sample (LCS) and LCS duplicate (LCSD) recoveries, matrix spike (MS) and matrix spike duplicate (MSD) recoveries, LCS/LCSD precision, MS/MSD precision, and duplicate precision are usually presented in the project QAPP. In some cases, however, the laboratory will be allowed to use internally derived control limits. If the control limits are specified in the QAPP, the validator should verify that the laboratory reports incorporate the required control limits. Failure to verify that the laboratory-reported control limits are those specified by the QAPP will cause QC discrepancies to be misidentified as conforming data points and conforming data points to be misidentified as discrepancies. In both cases, the data will not be evaluated against the requirements for precision and accuracy specified in the QAPP. This scenario can result in misqualified data and in additional validation effort. It can also result in the laboratory failing to identify a QC discrepancy and subsequently failing to perform required corrective action. Verifying that the correct control limits are being presented prior to beginning the validation effort is the best way to ensure that the reported results meet the precision and accuracy requirements established for the project. If discrepancies are noted, the laboratory project manager should be notified that the data reporting pages do not present the correct information and that the laboratory should ensure that all future deliverables conform to the requirements of the QAPP.

If required QC review elements or individual pages are missing from a laboratory data report, and the missing information is a result of an error in report compilation (such as a missing or illegible page), the validator should contact the laboratory project manager directly and request that the missing information be provided. If the missing information is to the result of a laboratory report generation convention (that is, the lack of a required data QC element is due to report design, not to an error in report compilation), the data validator should contact the HGL project chemist. The HGL project chemist will work with the laboratory project manager to ensure that any required information is provided to the data validators in alternative formats so that all QAPP-required QC elements can be reviewed.

3.3 DATA VALIDATION REPORTS

Data validation will be documented in a data validation report. Usually, data validation reports will be prepared for each analytical method and matrix reported in a single sample delivery group (SDG). For example, an SDG containing soil sample results for volatile organic compounds (VOCs), semivolatile organic compounds (SVOCs), pesticides, polychlorinated biphenyls (PCBs), and metals will have a separate data validation report produced for each of the analytical methods. Note that it is customary to combine the review of all metals results into a single report even if

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multiple analytical methods are used to generate the results; however, it is acceptable to subdivide metals data validation reports by method if the organization of the laboratory report makes this a more coherent way to present the results of the evaluation. The same is true for what are termed "wet chemistry" parameters.

In cases where individual project requirements conflict with the requirements of this SOP, the project requirements will take precedence. Any deviations from specified requirements, either of this SOP or project-specific, will be justified in the corresponding data validation report. Deviations from requirements will be sufficiently documented to allow the senior reviewer to evaluate whether such deviations are technically appropriate.

Example data report formats are presented in Attachment B. Note that the qualification conventions used in the example reports are based on the requirements of a specific project. The qualifiers assigned during the validation process should reflect the project's conventions.

3.4 PEER REVIEW

All data validation reports will be subject to a secondary review by either a peer or senior chemist assigned by the project manager. The peer reviewer will evaluate the data validation report against the contents of the data package to ensure the following applies:

- 1. The data validator has correctly applied the project requirements to evaluate and qualify the reported sample results.
- 2. The data validator has not overlooked any QC discrepancies present in the data package.
- 3. The validator has correctly associated any QC discrepancies with the correct analytes and analyses.
- 4. The assigned data qualifiers are complete and correct.
- 5. The data validator has not made "boilerplate" errors (that is, the inclusion of extraneous and incorrect information in the data report as a result of using another report as a template without removing inapplicable material).

A validation report that has not been reviewed will not be considered final.

3.5 SUBCONTRACTED DATA VALIDATION

The goal of subcontracted data validation is to generate a validated project dataset that is qualified in accordance with QAPP requirements and ready for HGL to upload into the project database. Subcontracted data validation will be performed in accordance with the individual firm's internal procedures and policies; however, the overall procedure must include pre-review, validation by qualified personnel, and peer or senior review of all data validation reports before delivery to HGL. All validation should be performed in accordance with the project QAPP and the scope of

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work provided by HGL. In most cases, the subcontracted validator will be responsible for providing qualified data electronically in a format that allows upload into HGL's project database (see Section 6.0), usually in the form of an Excel file. The validation firm will be responsible for all data entry, data entry QC, and removal of any residual laboratory-applied flags prior to delivery to HGL.

HGL will review data validation reports provided by third-party contractors in accordance with the procedures presented in Attachment F. The initial data validation reports provided by the contractor should be reviewed in depth by an HGL senior chemist as soon as possible to provide the data validator with timely feedback to guide ongoing validation efforts. The primary purpose of the HGL chemist senior review is to verify that the data validators understand the QAPP and project data quality requirements and are applying these requirements correctly when reviewing each data package. Data validation involves a large amount of professional judgment and there are multiple conventions that are technically valid. Therefore, a secondary purpose of the HGL senior chemist review is to ensure that the conventions HGL has selected are being used by the contractor in order to maintain consistency in evaluation and application of qualifiers. When it has been established that HGL's expectations are being met, subsequent data validation reviews can be streamlined to verify that the identified QC issues discussed in each validation report led to correct qualification of the associated sample results.

4.0 PERSONNEL

Data validation and review must be conducted by appropriately qualified and trained personnel.

4.1 ROLES, RESPONSIBILITIES, AND QUALIFICATIONS

4.1.1 HGL Project Staff

HGL project staff will be assigned in accordance with contract requirements and HGL's project management procedures. The following personnel have a wide range of responsibilities associated with their project titles; however, only the responsibilities applicable to the data validation process are discussed.

HGL Project Manager – Provides the data validation team with the information listed in Section 3.1, either directly or through a designee (such as a task manager). Works with HGL project and senior chemistry staff to identify appropriate personnel to conduct data validation and validation review activities for a project.

HGL Project Chemist – Provides guidance on analytical method requirements for sampling, preservation, and holding time requirements to field sampling teams. Assists the project manager in assigning data validation staff (see Section 4.1.2). Resolves issues that are not covered by the QAPP or other guidance documents. Ensures that laboratory performance is in accordance with HGL's project technical requirements. For projects with subcontracted data validation, the project

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chemist is responsible for reviewing data validation reports to verify that the data validation contractor is performing in accordance with the contract scope of work and the QAPP (see Appendix F).

HGL Senior Chemist – Provides overall direction to HGL's data validation program. Assists the project chemist in resolving issues that are not covered by the QAPP or other guidance documents. Assists the project chemist in ensuring that laboratory and validation contractor, if applicable, performance is in accordance with HGL's project technical requirements.

4.1.2 Data Validation Staff

Data validation staff includes data validators and peer reviewers who are assigned on an as-needed basis. Data validation staff can consist of qualified HGL personnel including chemists, geologists, environmental scientists, or other technical staff who have been trained in data validation by an HGL senior chemist or are judged by an HGL senior chemist to have sufficient experience in data validation. The qualifications and roles of data validation staff are described below.

HGL Data Validator – Should have at least a bachelor's degree in chemistry or other scientific discipline. The HGL data validator will perform data validation, communicate with the laboratory to resolve issues, and write the data validation reports. Data validation reports generated by an HGL validator with less than 1 year of experience should be reviewed by an HGL senior chemist.

HGL Peer Reviewer – Should have at least a bachelor's degree in chemistry or other scientific discipline and at least 2 years of data validation experience. Peer reviewers will perform a complete review of the findings of each data validation report against the associated laboratory data deliverable and determine if the validator has (1) addressed all QC issues affecting project data in accordance with the requirements of the project QAPP, (2) assigned the correct qualifiers to the reported data, (3) complied with project validation conventions, and (4) presented a clear description of the data quality issues affecting the reported data. Peer reviewers with less than 1 year of peer review experience shall be subject to approval by an HGL senior chemist before assignment.

Depending on the size of the project and staffing requirements, multiple data validators and peer reviewers may be assigned to a project; a data validator assigned to one laboratory deliverable may be a peer reviewer for another laboratory deliverable validation report. It is recommended, but not required, that the each project's project chemist be one of the HGL personnel assigned to perform data validation and peer review tasks for that project.

4.2 TRAINING REQUIREMENTS

HGL data validation staff should be trained directly by an HGL senior chemist. This training will preferably take place in person to allow for greater efficiency in instruction, evaluation, and feedback. Training will include validation of laboratory data reports followed by feedback and revision.

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5.0 PROCEDURES

Data should be reviewed and qualified in accordance with the project QAPP and validator judgment. The qualification guidelines presented in each OAPP are based on the project data quality objectives (DQOs) and will specify the level of data validation required to meet those DOOs. Level II and Level III are the most common levels of validation specified by project QAPPs. These levels will usually include only the examination of the information presented on laboratory-generated summary forms. This approach is generally sufficient to determine that the laboratory is following analytical method and project-specific requirements. On occasion, the review of specific raw data elements will be necessary to supplement the information that is present on summary reporting forms. Most HGL subcontracted laboratories are subject to intensive auditing procedures under the National Environmental Laboratory Accreditation Program (NELAP), the U.S. Department of Defense (DoD) Environmental Lab Accreditation Program (ELAP), and state accreditation bodies. These audits serve as verification that the laboratory's procedures for overall operations, analysis, data reduction, quality assurance, and data storage and retrieval are sufficiently rigorous and documented. As a result, Level IV data validation, which includes a detailed review of instrument raw data and laboratory records and provides the most rigorous evaluation of data quality, is rarely required for HGL's projects.

The specific procedures required to perform data validation vary greatly among data reports. The sources of variation include method QC requirements, client and regulatory requirements, laboratory-specific reporting conventions, and sample matrix. General guidelines for the evaluation of Level II QC elements and method-specific Level III QC elements are presented in Attachment C.

Both Level II validation and Level III validation can be supported by ADR software. A description of the ADR process and its integration into the data validation process is presented in Attachment D.

6.0 DATABASE QUALIFICATION

After the method-specific data validation reports for an SDG have been generated in accordance with Section 3.3 and reviewed in accordance with Section 3.4, the data qualifiers assigned by the validator are applied to electronic database output files. The procedures for data entry, review, and upload are presented in HGL QA/QC SOP, SOP No. 4.10. During what is referred to as the "100 percent QC stage" of this process, all residual laboratory-generated information flags that are not retained as the final qualification must be removed from each result. The only laboratory-generated flags that are retained are those that have been accepted as the final qualifier by the data validator. When data validation has been subcontracted, removal of residual laboratory flags will be the responsibility of the contractor prior to delivering qualified data files to HGL.

In some cases, projects will require the application of a reason code as well as a qualifier to validated results. In such cases, the HGL project chemist will develop a listing of reason codes,

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and these codes will be included in the data validation reports as an additional column in the data qualification table included in these reports. The HGL database manager will upload these reason codes into the database. Common reason codes are included in Attachment E.

7.0 SENIOR DATA RE-EVALUATION

When severe QC discrepancies are encountered, it may become necessary to reject associated data points. Rejected data points cause data gaps in the resulting dataset and may prevent that dataset from being able to be used to achieve project DQOs. Not all data gaps attributable to rejected results have an equal impact, however. Of special concern are (1) rejected results that affect a contaminant that has potential to be present at the subject site or (2) rejection of a large number of analytes in individual samples because of sample-specific or batch-specific QC issues.

If results are rejected in the initial data validation, the issue should be evaluated for referral to HGL's senior chemist for supplemental senior review. This review will include discussions with laboratory QA personnel, examination of raw data, and evaluation of the end use of the affected data. The review will evaluate the feasibility of replacing the R (reject) qualifier with a less severe qualifier. In some cases, removal of the R qualifier will not be technically justified and the affected results will remain rejected. In others, it may be determined that the affected results can be used to support decision making and the R qualifier will be replaced by a less severe qualifier. In all cases where HGL determines that rejection is not required, in contradiction to the requirements of the QAPP, the HGL senior chemist will document this judgment. This documentation should be made available to the client for review and approval, either in the form of technical memoranda or discussion in the associated project report.

8.0 REFERENCES

- U.S. Department of Defense Environmental Data Quality Workgroup, 2006. Department of Defense Quality Systems Manual for Analytical Laboratories, Final Version 4.2. October.
- U.S. Environmental Protection Agency (EPA), 2008a. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods SW-846. Third Edition, November 1986; Revision 1, July 1992; Revision 2, November 1992; Update II, September 1994, Update III, December 1996; Update IIIA, May 1999; Update IIIB, June 2005; and Update IV, January.
- EPA, 2008b. USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review. EPA-540-R-08-01. June.
- EPA, 2009. Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use. OSWER 9200.1-85; EPA-540-R-08-005. January.
- EPA, 2010. USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review. OSWER 9240.1-51; EPA-540-R-10-011. January.

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9.0 ATTACHMENTS

Attac	chment	A	Compo	onents	of I	Level	II,	III,	and	IV	Data	Revie	W:

Attachment B Example Data Validation Reports
Attachment C General Validation Guidelines

Attachment D Automated Data Review

Attachment E HGL Data Qualification Reason Codes

Attachment F Review of Subcontracted Data Validation Reports

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ATTACHMENT A Components of Level II, III, and IV Data Review

All Analytical Fractions	Level II	Level III	Level IV
Case narrative	X	X	X
Chain of custody	X	X	X
Sample receipt and log-in forms	X	X	X
Sample ID cross reference (HGL sample ID to laboratory sample ID)	X	X	X
Sample discrepancy reports, corrective action, and client communications	X	X	X
Holding times (preparation and analysis)	X	X	X
LCS/LCSD ⁽¹⁾ recoveries and precision	X	X	X
MS/MSD ⁽²⁾ recoveries and precision	X	X	X
Method blanks	X	X	X
Field blanks (trip, ambient, equipment, and material blanks)	X	X	X
Field duplicate precision	X	X	X
GC/MS Organic Analytical Fractions	Level II	Level III	Level IV
Surrogate recoveries	X	X	X
Instrument tuning		X	X
Instrument initial calibration		X	X
Second source calibration verification		X	X
Instrument continuing calibration verification		X	X
Internal standards		X	X
Chromatograms			X
Quantitation reports			X
Mass spectra			X
Calculations			X
Transcription			X
GC and HPLC Organic Fractions ⁽³⁾	Level II	Level III	Level IV
Surrogate recoveries	X	X	X
Instrument initial calibration		X	X
Second source calibration verification		X	X
Instrument continuing calibration verification		X	X
Degradation summary (organochlorine pesticides only)		X	X
Retention times		X	X
Confirmation		X	X
Chromatograms			X
Quantitation reports			X
Calculations			X
Transcription			X
Metals Fractions	Level II	Level III	Level IV
		X	X
Laboratory duplicate ⁽²⁾ precision	X	Λ	
	X X ⁽⁴⁾	X	X
Laboratory duplicate ⁽²⁾ precision Initial and continuing calibration blanks Instrument tuning		X X	X
Laboratory duplicate ⁽²⁾ precision Initial and continuing calibration blanks Instrument tuning Internal standards		X	
Laboratory duplicate ⁽²⁾ precision Initial and continuing calibration blanks Instrument tuning Internal standards Initial multipoint calibration ⁽⁵⁾		X X	X
Laboratory duplicate ⁽²⁾ precision Initial and continuing calibration blanks Instrument tuning Internal standards		X X X	X X

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ATTACHMENT A (continued) Components of Level II, III, and IV Data Review

Metals Fractions (continued)	Level II	Level III	Level IV
Initial and continuing calibration verification		X	X
Interference check sample results		X	X
Serial dilution results		X	X
Postdigestion spike recoveries		X	X
Recovery test recoveries (GFAA methods only)		X	X
Method of standard addition results		X	X
Interelement correction factors			X
Instrument raw data			X
General Chemistry Fractions	Level II	Level III	Level IV
Laboratory duplicate ⁽²⁾ precision	X	X	X
Method-specific QC checks ⁽⁷⁾	X	X	X
Initial and continuing calibration blanks	$X^{(4)}$	X	X
Initial multipoint calibration		X	X
Initial and continuing calibration verification		X	X
Method-specific instrument QC		X	X
Instrument raw data			X

- (1) LCSDs are not a requirement for any method or project; however, they are often provided by the laboratory. They will be reviewed similar to LCSs when available.
- (2) The analytical methods allow for metals and general chemistry precision to be evaluated either using MS/MSDs or laboratory duplicates at the laboratory's discretion. Often laboratories will provide both. The data validator will review all available QC data provided by the laboratory.
- (3) These methods use a second column or detector to confirm detected results. QC elements for both columns/detectors should be reviewed during the validation process.
- (4) The review of initial and continuing calibration blanks during a Level II review will vary on a project-by-project basis; this requirement will be specified in the OAPP.
- (5) Review of the initial multipoint calibration during Level III or Level IV validation is optional for ICP methods; if performed, the validator will review the associated results.
- (6) High- or low-level calibration verification is not required if initial multipoint calibration performed.
- (7) An example of method-specific QC checks is distillation checks for cyanide analysis.

Notes:

GC/MS = gas chromatography/mass spectrometry
GFAA = graphite furnace atomic absorption
HPLC = high-performance liquid chromatography

ICP = inductively coupled plasma LCS = laboratory control sample

LCSD = laboratory control sample duplicate

MS = matrix spike

MSD = matrix spike duplicate

QAPP = Quality Assurance Project Plan

QC = quality control

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ATTACHMENT B Example Data Validation Reports

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B.1 Example VOCs Data Validation Report

VOCs SW-846 Method 8260B Level III Review

Site: Hero Air Force Base	SDG #: ABC-1234	
Laboratory: TestGood Labs	Date: 08-05-08	
HydroGeoLogic, Inc. Reviewer: Joseph Vilain	Project: AF0555.01.02.03	
Peer Reviewer: Ken Rapuano (8.14.08)		

Client Sample ID	Laboratory Sample ID	Matrix	
MW123GW071108	ABC-1234-1	Water	
TRIP BLANK 52	ABC-1234-2	Water	

<u>Narrative and Completeness Review</u> - The case narrative and the data package were checked for completeness. No discrepancies noted.

Qualification: None required.

<u>Sample Delivery and Condition</u> – The samples arrived at the laboratory in acceptable condition, at proper temperature, and were properly preserved. Proper custody was documented.

Qualification: None required.

Holding Times – All samples were analyzed within the required holding time for preserved water samples.

Qualification: None required.

<u>Initial Calibration</u> – The initial calibration performed had acceptable mean RRFs for all SPCCs and %RSDs for all CCCs. SPCC bromoform was calibrated to a curve and no mean RRF was reported on the ICal summary; a mean ICal RRF of 0.1149 was reported on the CCal summary page. All target analytes calibrated to mean RRF had mean RRFs above 0.05 and %RSDs below 15%. All target analytes calibrated to curves had r² greater than 0.990. The second source verification standard met the control criteria.

Qualification: None required.

<u>Continuing Calibration</u> – The CCV standards had acceptable CCRFs for all SPCCs and %Ds for all CCCs. All target analytes had %Ds below 20.

Qualification: None required.

GC/MS Tuning – The samples analytical sequences were all performed within 12 hours of an acceptable GC/MS tune.

Qualification: None required.

<u>Surrogates</u> - All surrogate recoveries were within control limits specified in the QAPP.

Qualification: None required.

Laboratory Control Sample/Duplicate - All %R results LCS met control limits specified in the QAPP.

Qualification: None required.

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MS/MSD - Matrix spike/matrix spike duplicate analyses were not performed on this sample.

Qualification: None Required.

Internal Standards – All internal standards met area and retention time criteria.

Qualification: None required.

Method Blank - The method blank associated with the samples was free from contamination.

Qualification: None required.

Trip Blank - A trip blank, designated TRIP BLANK 52, was free from contamination.

Qualification: None required.

Equipment Blank - An equipment blank was not associated with this SDG.

Qualification: None required.

Field Duplicate - A field duplicate was not provided with this SDG.

Qualification: None required.

<u>Compound Quantitation</u> – Analyte non-detections were qualified U and reported as the LOD. These U flags were retained unless superseded by a more severe qualifier. Analytes detected between the DL and LOQ were reported as F qualified results by the laboratory. These F qualifiers were retained unless superseded by a more severe qualifier. Sample MW123GW071108 was diluted and reanalyzed at 10x dilution because of high concentration of vinyl chloride; the reporting limits were adjusted accordingly. Only the diluted vinyl chloride result was reported.

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Reason	
Validated	
Validated	
Lab	
Lab Value	
Analyte	N1 - 1.00
Sample	MW123GW071108

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B.2 Example Pesticides Data Validation Report

Organochlorine Pesticides

SW-846 Method 8081 Level III Review

Site: Hero Air Force Base	SDG #: ABC-1234
Laboratory: TestGood Labs	Date: 08-05-08
HydroGeoLogic, Inc. Reviewer: Joseph Vilain Peer Reviewer: Ken Rapuano (8.14.08)	Project: AF0555.01.02.03

Client Sample ID	Laboratory Sample ID	Matrix
MW123GW071108	ABC-1234-1	Water

 $\underline{\text{Narrative and Completeness Review}} - \text{The case narrative and the data package were checked for completeness.}} \ \ \underline{\text{No discrepancies noted.}}$

Qualification: None required.

<u>Sample Delivery and Condition</u> – The samples arrived at the laboratory in acceptable condition and at proper temperature. Proper custody was documented.

Qualification: None required.

Holding Times - All samples were extracted and analyzed within the required holding time.

Qualification: None required.

Initial Calibration – All target analyte %RSD values were less than 20%. The second source standard had %Ds less than 15%.

Qualification: None required.

<u>Continuing Calibration</u> – The continuing calibration verification standards bracketing the environmental samples in this SDG had all target analyte average %Ds below 15%.

Qualification: None required.

Surrogates - The recovery for all surrogates was within the acceptance limits specified in the QAPP.

Qualification: None required.

Retention Times - All retention times were within established retention time windows.

Qualification: None required.

<u>Laboratory Control Sample/Duplicate</u> – All target compound %Rs and RPDs were within the control limits specified in the QAPP in the LCS/LCSD.

Qualification: None required.

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MS/MSD - Matrix spike/matrix spike duplicate analyses were not performed on this SDG.

Qualification: None required.

Method Blank - The method blank was free from contamination.

Qualification: None required.

DDT/Endrin Breakdown Check - The DDT/Endrin breakdown standards had acceptable performance.

Qualification: None required.

Field Blanks - A field blank was not submitted with this SDG.

Qualification: None required.

Equipment Blanks - An equipment blank was not provided with this SDG.

Qualification: None required.

Field Duplicate - A field duplicate was not provided with this SDG.

Qualification: None required.

<u>Compound Quantitation</u> – Analyte non-detections were qualified U and reported as the LOD. These U flags were retained unless superseded by a more severe qualifier. Analytes detected between the DL and LOQ were reported as F qualified results by the laboratory. These F qualifiers were retained unless superseded by a more severe qualifier.

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	Reason	
	Validated Qualifier	
	Validated Value	
	Lab Qualifier	
	Lab Value	
icentrations in µg/L)	Analyte	No qualification required
Qualification Summary Table (all concentrations in µg/L)	Sample	MW123GW071108

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B.3 Example Metals Data Validation Report

Metals

SW-846 Method 6020A ICP-MS SW-836 Method 6010B ICP Mercury SW-846 7470A Level III Review

Site: Hero Air Force Base	SDG #: ABC-1234
Laboratory: TestGood Labs	Date: 08-05-08
HydroGeoLogic, Inc. Reviewer: Joseph Vilain Peer Reviewer: Ken Rapuano (8.14.08)	Project: AF0555.01.02.03

Client Sample ID	Laboratory Sample ID	Matrix
MW123GW071108	ABC-1234-1	Water

<u>Narrative and Completeness Review</u> – The case narrative and the data package were checked for completeness. It was stated in the narrative that batch QC analyses were performed on a sample associated with SDG ABC-1245. The narrative is in error; the associated ICP and ICP-MS QC sample data are reported in SDG ABC-1252.

Qualification: None required.

<u>Sample Delivery and Condition</u> – The samples arrived at the laboratory in acceptable condition, at proper temperature and were properly preserved. Proper custody was documented.

Qualification: None required.

Holding Times – All samples were analyzed within the required holding time for preserved water samples.

Qualification: None required.

Calibration - The ICVs and CCVs met the <10 % D (20% D for mercury) criterion.

Qualification: None required.

<u>ICP/MS Tuning</u> – The ICP/MS tune was <0.1 amu from the true value. Resolution and %RSD met acceptance criteria. The laboratory used 5% peak height to measure peak resolution, which is more stringent than the 10% peak height allowed by the QAPP.

Qualification: None required.

ICP Internal Standards – The environmental samples showed acceptable internal standard performance.

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<u>Low Level Sensitivity Check</u> – The low level calibration check had all target metals <20% D. No sensitivity check was provided for ICP-MS analyses (except copper and beryllium). It was noted that the samples in this SDG were analyzed in the same ICP-MS sequence as the site samples reported in SDG ABC-1252, and the results for sensitivity standard ABC-42690/18 shown in the SDG ABC-1234 run log are reported in SDG ABC-1252. Standard ABC-42690/18 had all target metals with <20% D. The laboratory also provided data for CRQL standards spiked at 2x the RL; all results met the laboratory control limits of 50-150%.

Qualification: None required.

Method and Calibration Blanks – The ICP and ICP/MS method blank was contaminated with several metals. The calibration blanks bracketing sample analyses were also contaminated. The mercury method blank and calibration blanks were free from contamination. The contaminations are listed below in tabular form.

Metal	Contamination Point	Highest	Artifact Level
Wietai	Contamination Fourt	Contamination (ug/L)	(ug/L)
Calcium	MB	35	175
Antimony	MB, CCB12	2.1*	10.5
Arsenic	CCB12	0.33	1.65
Beryllium	CCB11	0.20	1.0
Cadmium	CCB12	0.21	1.05
Lead	MB, ICB, CCB11	0.17	0.85
Silver	CCB11	0.14	0.70
Thallium	ICB, CCB12	1.3	6.5
Zinc	MB	6.3	31.5

^{*}Result above the MQL; no corrective action was taken.

Detected results below the artifact levels generated by positive blanks are considered analytical artifacts and are qualified B.

Sample results associated with method blank contamination (but not ICB or CCB contamination) were reported qualified B by the laboratory regardless of whether the sample concentration was greater or less than the associated artifact level. All laboratory applied B flags should be removed unless the final qualifier for that result is B.

Qualification: Antimony and thallium are qualified B. Remove all other B flags applied by the laboratory.

Interference Check Sample – The ICSA and ICSAB spiked analytes met the <20% D criteria, with the exception of selenium (121%R) in run 42690/15; however, this ICSA did not bracket the sample analysis and no qualification is necessary. All unspiked analytes were below the RL.

Qualification: None required.

ICP Serial Dilutions/Post Digestion Spike Samples – As was noted in the narrative, ICP and ICP-MS batch QC was run on a site sample from another job (the correct SDG is ABC-1252). The serial dilution and PDS associated with method 6010B were performed using site sample MW125GW071208. The serial dilution and PDS associated with method 6020 were performed using site sample MW132GW071208. Serial dilution analysis results were within specification for target analytes present in the parent samples at greater than 50x MDL (6010B) or 100x MDL (6020). Post digestion spike results were within specification for all analytes.

Qualification: None required.

Laboratory Control Sample - The %R results for the LCS met control limits.

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MS/MSD – As was noted in the narrative, ICP and ICP-MS batch QC was run on a site sample from another job (the correct SDG is ABC-1252). The MS/MSD associated with method 6010B was performed using sample MW125GW071208. The MS/MSD associated with method 6020 were performed using sample MW132GW071208. The MSD had low %Rs for calcium and iron; these results are not applicable because the sample concentration was >4x the spiking level. The zinc %Rs were in control; however, the RPD was 29%, which is above the control limit. The detected zinc result should be qualified J.

Qualification: The zinc result is qualified J.

<u>Laboratory Duplicate Sample</u> - A laboratory duplicate was not prepared from this SDG.

Qualification: None required.

Field Duplicate - A field duplicate was not provided with this SDG.

Qualification: None required.

Equipment Blank - No equipment blank was associated with this SDG.

Qualification: None required.

<u>Compound Quantitation</u> – Analyte non-detections were qualified U and reported as the LOD. These U flags were retained unless superseded by a more severe qualifier. Analytes detected between the DL and LOQ were reported as F qualified results by the laboratory. These F qualifiers were retained unless superseded by a more severe qualifier.

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hg/L)
concentrations in
rable (all
Summary
Qualification

o ume o	Analydo	onley de l	l ah Oualifier	Validated Value	Validated	Reason	
ald line	and and	במם אמותם		validated value	Qualifier	Code	
	Calcinm	150000	В	150000			
	Antimony	0.62	FB	0.62	В		
MW123GW071108	Lead	2.8	В	2.8	-		
	Thallium	98.0	ш	0.86	В		
	Zinc	84	В	84	٦		

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B.4 Example General Chemistry Data Validation Report

Wet Chemistry

SW-846 Methods 9014 (Cyanide) Level III Review

Site: Hero Air Force Base	SDG #: ABC-1234
Laboratory: TestGood Labs	Date: 08-05-08
HydroGeoLogic, Inc. Reviewer: Joseph Vilain Peer Reviewer: Ken Rapuano (8.14.08)	Project: AF0555.01.02.03

Client Sample ID	Laboratory Sample ID	Matrix
MW123GW071108	ABC-1234-1	Water

 $\underline{\text{Narrative and Completeness Review}} - \text{The case narrative and the data package were checked for completeness.} \\ \text{No discrepancies noted.}$

Qualification: None required

<u>Sample Delivery and Condition</u> – The samples arrived at the laboratory in acceptable condition, at proper temperature and were properly preserved. Proper custody was documented.

Qualification: None required.

Holding Times - All samples were analyzed within the required holding time.

Qualification: None required.

<u>Calibration</u> – The initial calibration associated with the environmental sample in this SDG met acceptance criteria. The ICVs met the %D<10% criterion. The continuing calibration verifications bracketing sample analysis also met the %D<10% criterion. The cyanide high and low distilled standards met %R criteria.

Qualification: None required.

Method and Calibration Blanks - The method blank and calibration blanks were free from contamination.

Qualification: None required.

Laboratory Control Sample - The %R met the control limits specified in the QAPP.

Qualification: None required.

MS/MSD – Matrix spike/matrix spike duplicate analyses were not performed on this SDG.

Qualification: None required.

Field Duplicate - A field duplicate was not provided with this SDG.

Qualification: None required.

Equipment Blank - An equipment blank was not provided with this SDG.

Qualification: None required.

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 $\underline{\textbf{Compound Quantitation}} - \textbf{Analyte non-detections were qualified U and reported as the MDL}. \ \, \textbf{These U flags were retained unless superseded by a more severe qualifier}.$

Qualification: None required.

Qualification Summary Table (all concentrations in mg/L)

Sample	Analyte	Lab Value	Lab Qualifier	Validated Value	Validated Qualifier
MW123GW071108	No qualification required				

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ATTACHMENT C General Data Validation Conventions

1.0 INTRODUCTION

The general conventions presented below describe the evaluation and qualification process applied to project data undergoing a Level II or Level III data validation. The data validator should always use the Quality Assurance Project Plan (QAPP) as the primary source for project-specific validation requirements. Where the general conventions presented below conflict with the requirements presented in the QAPP, the QAPP requirements should take precedence. Situations that are not covered by the project QAPP or by the general conventions should be referred to a HydroGeoLogic, Inc. (HGL) senior chemist for resolution.

2.0 SENSITIVITY LIMITS

The principal reasons for assigning data qualifiers is the magnitude of detected results relative to the associated sensitivity limits and the conventions for reporting nondetected results. There are two principal conventions for establishing sensitivity limits, the conventions originally established by the U.S. Environmental Protection Agency (EPA) to support the Contract Laboratory Program (CLP) and the conventions established by the U.S. Department of Defense (DoD). Both are in common use and are described below. Table C.1 presents the DoD terms, their definitions, and the corresponding EPA terms that are also in common usage.

Table C.1
Sensitivity Limit Definitions⁽¹⁾

Sensitivity Limit Term	Definition	Corresponding EPA Terms
Detection limit	The smallest analyte concentration that can be	Method detection limit (MDL)
(DL)	demonstrated to be different from zero or a	
	blank concentration at the 99% level of	
	confidence. At the DL, the false positive rate	
	(Type I error) is 1%.	
Limit of	The smallest amount or concentration of a	
detection	substance that must be present in a sample in	
(LOD)	order to be detected at a high level of	
	confidence (99%). At the LOD, the false	
	negative rate (Type II error) is 1%.	
Limit of	The lowest concentration that produces a	Reporting limit (RL)
quantitation	quantitative result within specified limits of	Quantitation limit (QL)
(LOQ)	precision and bias. For DoD projects, the LOQ	Practical quantitation limit (PQL)
	shall be set at or above the concentration of the	Method quantitation limit (MQL)
	lowest initial calibration standard.	Contract-required detection limit (CRDL)
		Contract-required quantitation limit (CRQL)

⁽¹⁾ Terms and definitions are from Appendix B of the DoD Quality Systems Manual for Environmental Laboratories, version 4.2 (October 2010).

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2.1 EPA SENSITIVITY LIMIT CONVENTIONS

The EPA convention involves setting a concentration limit above which analytical results are considered to be of sufficient quantitative significance to be reported without qualification (unless affected by quality control [QC] issues. In practice, this limit is established at or above the low point on the calibration curve for each target analyte. A variety of terms has been applied to this limit, including reporting limit (RL), practical quantitation limit (PQL), and method quantitation limit (MQL). EPA's CLP uses the term contract-required quantitation limit (CRQL) for organic results and contract required detection limit (CRDL) for inorganic results. Results between the MDL and RL are reported as detections qualified as estimated as a result of being below the calibrated range. Results below the MDL are considered nondetected results and are reported as the numerical value of the MDL or the RL (depending on project-specific requirements) qualified U.

For many of HGL's DoD projects, the EPA sensitivity limit conventions have been superseded by the DoD conventions described in Section 2.1.2; however, projects performed for non-DoD clients will still use the EPA conventions. Older DoD projects with existing basewide QAPPs also may retain the use of this convention to maintain comparability with the existing project dataset.

2.2 DOD SENSITIVITY LIMIT CONVENTIONS

The current DoD sensitivity limit conventions were introduced in version 4 of the Quality Systems Manual (QSM) in April 2009. The QSM established a three-tiered system of detection limit (DL), limit of detection (LOD), and limit of quantitation (LOQ). The QSM provides definitions for all these terms; however, in practical applications, the DL and LOQ are used in an analogous fashion as the MDL and RL, respectively, are used in the EPA sensitivity conventions. Results between the DL and LOQ are reported as detections qualified as estimated due to being below the calibrated range. The LOD term was introduced in the QSM and corresponds to the lowest level that can be present in a sample and have a 95 percent probability of being detected in that sample. In the DoD conventions, results below the DL are considered nondetected results and are reported as the numerical value of the LOD qualified U.

3.0 DATA QUALIFIERS

Each validated result consists of three components: (1) a numerical value that corresponds to a concentration, (2) a data qualifier, and (3) the concentration units. The concentration can correspond to a detected value or to a proxy value used for nondetected results in that is assigned accordance with the conventions presented in the project QAPP. The data validation process generally focuses on the application of the appropriate data qualifier on each result. Some projects will require a change to the numerical concentration presented under specific circumstances, but this is not common (see Section 3.2.4).

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Data qualification indicates that an analytical result falls into one of three broad categories: (1) usable; (2) usable but estimated; and (3) unusable. The validation conventions presented below do not present specific qualification requirements. The qualifiers to be used for a project will be defined in that project's QAPP. The allowed final data qualifiers will be defined depending on the client and the regulatory body that will be the final recipients of the data. Descriptions of commonly applied data qualifiers are presented below, but the data validator must use the qualification requirements specified in the QAPP for each project.

3.1 LABORATORY-APPLIED FLAGS

In some cases, data points may be reported by the laboratory with one or more informational flags, such as an alphanumeric code or a symbol. These flags are not considered valid qualifiers and should be automatically removed from all affected data points, with the exceptions noted in Sections 3.2.2, 3.2.4, and 3.3.1 below. In some cases, the laboratory-applied informational flag will mimic a valid final qualifier, but may or may not be applicable as the final qualifier. In such cases, the validator's discussion of the effect of a QC discrepancy on the associated results should also include a discussion of whether laboratory-applied flags that mimic a valid qualifier should be retained, deleted, or altered. All residual laboratory-applied flags that are not accepted as the final qualifier by the data validator must be removed from the electronic data at what is referred to as the "100 percent QC stage" of data upload and incorporation into the project database (see Section 6.0 of the standard operating procedure [SOP]).

Example: A laboratory uses a "B" flag to indicate that a metals result is below the calibrated range, but "B" is also a project-specific valid final qualifier used to indicate the validator's judgment that the affected result is an artifact. In some cases, the B flag applied by the laboratory for one reason will correspond to the final qualifier assigned for a different reason. In other cases, it will not. The validator should indicate which results reported with a B flag by the laboratory will have the B retained as the final qualifier and which results should have the B flag removed or replaced with other applicable final qualifier.

3.2 QUALIFICATION OF DETECTED RESULTS

3.2.1 Detected Results not Requiring Qualification

Results that are detected within the calibrated range of the instrument and which are not associated with a QC discrepancy are almost universally accepted by the validation process as the numerical value of the concentration (with appropriate units) and without any data qualifier.

3.2.2 Detected Results below the Calibrated Range

Detected results with concentrations greater than the DL but below the LOQ (corresponding to the lower limit of the calibrated range of the instrument) are considered to be estimated results

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by default. Laboratories report such results with an informational flag to indicate that the result is below the calibrated range. This informational flag is most often a "J" or an "F"; these flags correspond to commonly used final qualifiers that are applied to such results. When the laboratory assigns a flag that corresponds to the project qualification convention, the assigned flag can be accepted as the final qualifier by the validator if no other qualification is required for a QC issue. In other cases, the validator will need to specify that, absent any other qualification, results reported with the laboratory's default flag for a result below the LOQ will need to be globally changed to the project-specific qualifier.

Example: A laboratory reports detected results below the LOQ with an F flag, which is also the appropriate final qualifier for such results unless superseded by a more severe qualifier. The validator should state that these flags are accepted as the final qualifier unless otherwise noted in the validation report. Conversely, if the laboratory reports detected results below the LOQ with a J flag and the project requires such results to have an F qualifier, the validator should note that the laboratory-applied J flags should be changed to F qualifiers, unless superseded by a more severe qualifier.

3.2.3 Detected Results Requiring Qualification as Estimates

Detected results affected by QC issues will be qualified as estimated values as required by the project validation guidelines. The most common qualifier used to indicate an estimated result is "J." Some projects will use alternative qualifiers if the overall direction of bias can be determined. These alternative qualifiers can include "J+" or "K" if the bias is high, or "J-" or "L" if the bias is low. Some projects will also include an "M" qualifier to distinguish results that are considered estimated because of a matrix effect from those that are considered estimated due to a QC issue.

3.2.4 Detected Results Requiring Qualification as Artifacts

One of the goals of data validation is to determine if detected concentrations of analytes reported in samples are representative of site conditions. Detected concentrations reported by the laboratory that are artifacts of the sampling, shipping, storage, preparation, and analytical processes that the sample undergoes are not representative of the site and must be identified by the validator. The most common procedure to identify results as artifacts is to apply the qualification of "U" or "B."

In addition to being used to identify artifacts under some conventions, the U qualifier is almost universally used to identify nondetected results (see Section 3.3.1). When the U qualifier is also used for identifying artifacts, the final qualifier will not allow the data user to determine whether the analyte in question is a nondetection or was determined to be an artifact. However, artifacts are treated in the same fashion as nondetections for most end uses of analytical data, so in practice this convention does not introduce unacceptable ambiguity into the final result. The quantitated value associated with the U qualifier assigned to an artifact can be the originally reported detected value, the LOD, or the LOQ (or equivalent), depending on

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the data reporting conventions presented in the project QAPP. For projects using the DoD sensitivity limit conventions, results qualified U as artifacts that have a concentration that exceeds the LOD but are lower than the associated LOQ will receive a final qualifier of UJ.

If the B qualifier is used to identify artifacts, then the associated quantitated value will always be the originally reported detected value.

3.2.5 Rejection of Detected Results

Most data qualification conventions will avoid rejection of detected results unless severe QC deficiencies are identified. Detected results with extreme high or low bias, compromised by severe discrepancies in sample collection or shipment, or that were generated while the analytical system was unacceptably compromised will not be of sufficient quality to be incorporated into a quantitative risk assessment. In some cases, however, data points rejected in accordance with the validation protocols may have limited usability.

Example: A detected result is associated with a severe low bias but the result is greater than the screening level for the site. This result could be used to determine if that compound were a contaminant of concern at the site. It could also be used to determine that the result was greater than a compliance level. However, the numerical value is too compromised to be able to be incorporated into the quantitative determination of risk at the site.

Rejected detected results are qualified R. When detected results are rejected, the quantitated value associated with the final qualifier is the detected value reported by the laboratory.

3.3 QUALIFICATION OF NONDETECTED RESULTS

3.3.1 Nondetected Results not Requiring Qualification

Nondetected results receive a final qualifier of U in almost every data qualification convention. Depending on the requirements of the QAPP, the quantitated value associated with the U qualifier can either be the DL (or equivalent), the LOD, or the LOQ (or equivalent). The reporting conventions to be used for each project should be included in the project QAPP and should be confirmed with the laboratory prior to generating project results. For most projects, a large majority of the reported laboratory results will be nondetections. Ensuring that the laboratory will report nondetected data flagged U using the same protocols as are required for the final U qualification will allow the data validator to retain the laboratory flags unchanged.

Some laboratories report nondetected results as "ND" or as "<#" where # can be the DL (or equivalent), LOD, or LOQ (or equivalent). The data validation report should indicate that such results are considered to be the equivalent of results qualified U according to the project conventions, unless superseded by a more severe qualifier.

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3.3.2 Nondetected Results Requiring Qualification as Estimated

Nondetected results affected by QC issues will be qualified as estimated values as required by the project validation guidelines. The most common qualifier used to indicate an estimated result is the combination qualifier "UJ." Some projects will use alternative qualifiers if the overall direction of bias can be determined. These alternative qualifiers can include "UJ-" or "UL" if the bias is low. Nondetected results are not considered to be affected by high bias. As with nondetected results not requiring qualification, the quantitated value associated with the qualified result can be the DL (or equivalent), the LOD, or the LOQ (or equivalent), depending on the project conventions for reporting nondetected results.

3.3.3 Rejection of Nondetected Results

Nondetected results are generally rejected under more circumstances than detected results. This is because most projects consider a Type II (false negative) error to be a more severe error than a Type I (false positive) error. Rejected nondetected results are qualified R. As with nondetected results not requiring qualification, the quantitated value associated with the qualified result can be the DL (or equivalent), the LOD, or the LOQ (or equivalent), depending on the project conventions for reporting nondetected results.

3.4 QUALIFICATION OF EXCLUDED RESULTS

In cases where multiple analysis results are reported for a sample as a result of dilution or reanalysis, all analyses are to be reviewed. Based on the body of QC data, the validator should select one definitive result for each analyte in each sample, and all other results for that analyte in that sample shall be denoted as superseded by applying an X qualifier. Clearly indicating results that are not to be used with an X assists in managing data for report preparation and database submittal. Results that receive an X qualifier do not need to be further validated or qualified; however, the validation narrative should include the rationale for selecting the definitive result. Results receiving an X qualifier should be included in the data qualification table in each validation report, with the analysis receiving the qualification clearly differentiated from the other analyses performed on the same sample. Where large categories of results in a sample analysis receive an X qualifier, the X qualification may be noted for the class of results (for example, "All nondetections") instead of as an analyte-byanalyte listing. X qualification may result in the data for the full analyte list for a particular sample being composed of results from multiple analyses. For example, in an original analysis/diluted analysis pair, all analytes in the original analysis are considered definitive with the exception of those analytes that exceeded the calibrated range, which are reported from the diluted analysis.

The preferred procedure for applying X qualifiers is to append the X qualifier to the laboratory-applied informational flags rather than replacing the laboratory-applied flags with X. This procedure will preserve the information provided by the laboratory should the X qualification decision be revisited at a future time. The quantitated value associated with X

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qualified results is the quantitated value associated with the original detected or nondetected result reported by the laboratory.

3.5 RESULTS WITH MULTIPLE APPLICABLE QUALIFIERS

Some results may be affected by more than one QC discrepancy. In such cases, the final qualifier applied to each result is the highest priority qualifier as defined by the project QAPP.

When "U" is used the qualifier to denote an artifact, the validator should treat the associated result as a detection when evaluating additional qualification for other QC issues.

Example: A result is determined to be an artifact and the conventions call for that result to be qualified U. Another QC issue also affects that result, and the qualification conventions call for a detected result to be qualified J and a nondetected result to be qualified R. The validator should apply UJ as the final qualifier instead of R to any affected results that were reported as detections but are qualified U as a result of being considered an artifact.

4.0 LEVEL II QC ELEMENTS

The following are general guidelines for reviewing the QC elements identified as Level II QC elements in Attachment A. Final qualification will be applied in accordance with the QAPP.

4.1 CASE NARRATIVE

Qualification is usually not required based on the results of the case narrative; however, the validator should review the narrative prior to beginning validating the data package. The narrative can assist in identifying QC issues, describe corrective action or causes for QC discrepancies, describe sample receipt discrepancies, and indicate any special client instructions for the sample analyses. In the data validation report, the validator should include any items of note that were in the narrative, as well as indicate if there were any errors or omissions in the laboratory narrative.

4.2 CHAIN OF CUSTODY

Review the chain of custody (CoC) form and verify that there are no discrepancies. Some general issues can include difficult-to-read sample IDs, crossed-out items, incorrect analyses requested, incorrect or missing time of collection, and missing or incorrect preservative information. The laboratory also may indicate additional information on the CoC form such as special client requests, sample receipt temperature, and samples added or deleted from those requested on the chain. Generally, results are not qualified based on the CoC form alone; however, this information can be useful to the validator.

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4.3 SAMPLE RECEIPT AND LOG-IN FORMS

This form should be checked for discrepancies in sample temperature and sample preservation; discrepancies between the sample labels and the CoC forms; missing, broken, or damaged bottles; and bubbles in containers that should have zero headspace. Results may be qualified based on sample receipt and condition.

Some methods, such as metals and volatile organic compounds (VOC), allow for alternatives if preservation requirements are not met. Aqueous VOC samples are considered to be acceptable if bubbles in the vials are less than "pea-sized" (defined as approximately ¼ inch or 6 mm).

Although it is good practice to ship all samples iced, temperature discrepancies are less likely to affect persistent organic compounds like polynuclear aromatic hydrocarbons (PAHs), pesticides, and polychlorinated biphenyls (PCBs); temperature discrepancies should have minimal to no effect on metals samples. If the samples were delivered to the laboratory by courier on the same day they were collected, the samples may not have had enough time to chill to the acceptance range (0 to 6°C). In such cases, the sample temperature is considered to be compliant if the samples arrived at the laboratory iced and were refrigerated on arrival.

4.4 SAMPLE ID CROSS REFERENCE

Review the laboratory listing of HGL sample IDs against the CoC form. Common errors involving letter/numeral substitutions include "0" and "O" or "D"; "5" and "S"; "6" and "G"; and "8" and "B." Another common error is inconsistencies in incorporating dashes in sample IDs.

Another common error occurs at sample login when the parent sample and the requested matrix spike (MS) and matrix spike duplicate (MSD) samples are submitted in using an ID format that inserts "MS" and "MSD" into a long string of alphanumeric characters: "PARENTSAMPLEID," "PARENTMSSAMPLEID," and "PARENTMSDSAMPLEID." When there is no clear indication that a sample is an MS or an MSD sample, the laboratory log-in department may not notice that the sample IDs are indicating an MS or MSD, causing these samples to be logged in as "normal" samples. The result is that instead of results for parent sample and an MS/MSD pair, the samples are analyzed as a sample triplicate. In such cases, the laboratory log-in department should be notified to be alert for such sample IDs, and the HGL project manager should be alerted that more explicit instructions should be provided to the laboratory when submitting MS/MSDs.

4.5 HOLDING TIMES

The holding times for preparation and analysis for each analytical method should be presented in the project QAPP. HGL's general convention is to measure holding times using both date

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and time; this convention should be included both in HGL's laboratory statement of work and project QAPP.

Example: An aqueous semivolatile organic compounds (SVOC) sample collected at 10:00 a.m. on May 1 would have a preparation holding time of 7 days, which would expire at 10:00 a.m. on May 8.

The validator should be aware that time zone difference and daylight savings time need to be accounted for when evaluating holding time. Also, some sampling teams assign a "dummy" sample collection time (such as "1200") to field duplicate samples. Before qualifying field duplicate sample results for a holding time exceedance of less than a day, the validator should verify the actual sample collection time with the field team.

The validator has some discretion to consider a holding time exceedance to be nominal and determine that qualification is not necessary. This discretion should be used when the holding time discrepancies are isolated instances. Some laboratory reporting forms only report date and not time for sample preparation. If no time is available, the data validation should calculate the holding time based on the day and note this in the data validation report.

4.6 LCS/LCSD RECOVERIES AND PRECISION

As discussed in Section 3.2 of the SOP, the validator should verify that the control limits reported by the laboratory match those required in the project QAPP. Note that laboratory control sample duplicates (LCSD) are not a QC element required by any analytical methods; however, reporting an LCSD in association with a laboratory control sample (LCS) is a common laboratory practice. When LCSDs are reported, the accuracy performance should be evaluated in the same manner as the associated LCS, and discrepancies in either the LCS or LCSD should be considered grounds for qualifying associated data. In some cases, however, the validator can consider acceptable performance in the LCS or LCSD as a mitigating factor and reduce the severity of the data qualifier applied to associated results for a discrepancy in the other member of the LCS/LCSD pair. The decision to reduce the severity of the data qualifier in this instance should be discussed in the data validation report.

LCSs (and LCSDs) should be spiked with the full list of target analytes unless the QAPP specifically allows for the use of a shorter list. The exception is in the analysis of PCBs. As a result of the existence of multiple overlapping peaks in the spectrum of each individual PCB congener, PCBs LCSs are spiked with a mixture of PCB-1016 and PCB-1260. Generally, discrepancies shown by PCB-1016 are considered to affect PCBs 1016, 1221, and 1232; and discrepancies shown by PCB-1260 are considered to affect PCBs 1242, 1248, 1254, and 1260.

LCS/LCSD recoveries that are above the acceptance limits are usually considered not to affect nondetected results. In cases of extremely high recoveries (approaching 200 percent or greater) the validator should consider whether an analytical system problem has occurred. If the cause for abnormally high recoveries is not noted in the case narrative, the validator should contact

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the laboratory and request an explanation for such anomalies. In some cases, such discrepancies can be traced to accidental double-spiking and the recoveries will meet acceptance criteria when calculated using the actual spiked concentration. However, the validator should consider the qualification of nondetected results associated with unusually high recoveries if the underlying cause indicates a problem in the analytical system.

When LCS/LCSD precision (the reported relative percent difference [RPD]) does not meet the requirements for an analyte, detected results for the affected analyte should be qualified in the associated samples. Nondetected results generally do not require qualification for LCS/LCSD precision discrepancies.

4.7 MS/MSD RECOVERIES AND PRECISION

The evaluation of MS/MSDs is generally the same as the evaluation performed on LCSs and (if performed) LCSDs. Given that MS/MSDs are intended as verification that the laboratory can detect target analytes in the project-specific sample matrix, only MS/MSD analyses performed on HGL-collected samples from the same site (or installation) are considered applicable to the sample results in a sample delivery group (SDG). Laboratories often report MS/MSD results for nonsite samples as batch control. The presence of these analyses should be noted in the validation narrative, but the results reported from these batch control analyses are not used to qualify data.

MS/MSD discrepancies in samples that have concentrations of the affected target analytes greater than 4 times the spiked concentration are not considered applicable. Dilution should reduce or eliminate matrix effects and MS/MSD discrepancies in cases where the MS and/or MSD were diluted require some interpretation on the part of the reviewer to determine whether there is actually a matrix effect or whether some other factor is contributing to the discrepancy. In cases where MS/MSD recoveries are calculated from spike recoveries that are above the calibrated range, the reviewer should evaluate whether any discrepancies are a result of matrix effects or are a result of the inherent unreliability of such results.

MSs (and MSDs) should be spiked with the full list of target analytes unless the QAPP specifically allows for the use of a shorter list. The exception is in the analysis of PCBs. Because of the existence of multiple overlapping peaks in the spectrum of each individual PCB congener, PCBs MS/MSDs are spiked with a mixture of PCB-1016 and PCB-1260. Generally, discrepancies shown by PCB-1016 are considered to affect PCBs 1016, 1221, and 1232; and discrepancies shown by PCB-1260 are considered to affect PCBs 1242, 1248, 1254, and 1260.

Note that in some cases, the laboratory will report MS/MSD results from a different SDG as batch control without the client sample ID. When a batch control MS/MSD is reported, the validator should use the laboratory sample ID to confirm whether the MS/MSD is actually from a site sample reported in a different SDG or from a nonsite sample. If the MS/MSD is from a site sample, it will be considered applicable to associated results. If the MS/MSD

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cannot be associated with a site sample, the results should be noted but no qualification should be applied unless the underlying cause of the discrepancy is suspected to be a problem with the analytical system.

Some laboratories compare the concentrations detected in the MS and the MSD to calculate precision rather than comparing the percent recoveries. This convention can lead to the resulting RPDs being an incorrect representation of the analyte-specific precision. If the expected concentration in the MS is different from the expected concentration in the MSD, calculation of the RPD using a direct comparison of the detected concentrations is not relevant. The validator should verify that the RPDs reported for MS/MSD results are calculated using the percent recoveries or that the expected concentration in the MS is the same as in the MSD. If the RPDs are calculated using noncomparable results, the validator should contact the laboratory and request reporting pages with the calculations performed using percent recoveries. If this information cannot be produced by the laboratory, the validator will have to perform these calculations.

For some methods, it is permissible to analyze a single MS as a check for accuracy and use a laboratory duplicate as the check for precision. Laboratory duplicate evaluation is discussed under field duplicates (Section 4.10). If the laboratory performs both an MSD and a laboratory duplicate, both should be evaluated and used to qualify associated results.

The qualification of results for MS/MSD discrepancies is project- and method-specific. Generally, inorganic and wet chemistry MS/MSD results are considered to be associated with all environmental samples in the same preparation batch and organic MS/MSD results are considered to be associated only with the parent sample.

The QAPP should include additional instructions for evaluating and qualifying results based on MS/MSD discrepancies. Nondetected results generally do not require qualification for MS/MSD precision discrepancies. MS/MSD recoveries that are above the acceptance limits are usually considered not to affect nondetected results. In cases of extremely high recoveries (approaching 200 percent or greater) that are not attributable to native analyte concentration or matrix effects, the validator should consider whether an analytical system problem is occurring. If the cause for abnormally high recoveries is not noted in the case narrative, the validator should contact the laboratory and request an explanation for such anomalies. In some cases, such discrepancies can be traced to accidental double-spiking and the recoveries will meet acceptance criteria when calculated using the actual spiked concentration. However, the validator should consider the qualification of nondetected results associated with unusually high recoveries if the underlying cause indicates a problem in the analytical system.

4.8 METHOD BLANKS

HGL's QAPPs list acceptance criteria for method blanks. These acceptance criteria are the levels above which blank contamination necessitates that the laboratory performs corrective

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action. However, *all* method blank concentrations that are greater than the associated DL or have a negative concentration with absolute value greater than the associated DL should be used to qualify the associated sample results. The data validator should note any concentrations of target analytes detected in method blanks that are greater than the associated acceptance limits, including metals method blanks showing negative concentrations with absolute value greater than the acceptance limits.

Target analyte concentrations detected in method blanks should be multiplied by 5; this calculated value is called the artifact threshold. Concentrations of these analytes in associated samples that are less than the artifact threshold are considered artifacts and are qualified in accordance with the QAPP.

Concentrations of common laboratory contaminants are multiplied by 10 instead of 5 to determine the artifact threshold. Common laboratory contaminants for VOCs include methylene chloride, acetone, and 2-butanone (methyl ethyl ketone). Common laboratory contaminants for SVOCs are the phthalate esters.

When comparing method blank action levels to sample concentrations, the artifact threshold should be adjusted to account for sample-specific information, including percent moisture, subsample size, and dilution factor. Often, the easiest way to determine a sample-specific adjustment is to compare the LOQ of a target compound in the sample to the LOQ for that compound in the method blank.

Example: The method blank artifact threshold for toluene is calculated to be 4.3 micrograms per kilogram (μ g/kg). The toluene LOQ is 5 μ g/kg in the method blank and 7.4 μ g/kg in sample ABC123. The sample-specific action level for toluene is 4.3 x (7.4/5) μ g/kg = 6.4 μ g/kg.

In most cases, it will be readily apparent that a result is above or below an artifact threshold. In practice, this sample-specific adjustment is necessary for only a minority of comparisons.

4.9 FIELD BLANKS

Field blanks are evaluated in a similar manner as method blanks (Section 4.8). Two main differences are (1) the artifact threshold calculated from concentrations in field blanks is *not* adjusted for sample-specific factors; and (2) most field blanks are aqueous and conversion to equivalent solid units is not straightforward for some analytical methods.

When evaluating the effect of aqueous field blank results on associated aqueous field samples, the artifact threshold associated with field blank contamination is 5 times the concentration

¹ Note that the term "action level" was previously used to describe this value; the use of the term action level is discouraged because that term is also used in site characterization and has a different meaning when used in that context.

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detected in the blank (10 times the concentration in the case of common laboratory contaminants). When evaluating the effect of aqueous field blank results on associated solid matrix field samples, the field blank results must first be converted to the equivalent solid concentration.

4.9.1 Water-to-Soil Conversion for Organic Extraction Methods

Aqueous field blank results for organic extraction methods can generally be converted to solid units by comparing the ration of the aqueous LOQs to the LOQs reported in the solid matrix method blanks.

Example: A rinse blank has a detected result of 7.8 micrograms per liter (μ g/L) for diethyl phthalate. The aqueous LOQ is 10 μ g/L and the solid LOQ is 330 μ g/kg. The diethyl phthalate result in the rinse blank is the equivalent of a result of 257.4 μ g/kg (7.8 x 330/10). Because diethyl phthalate is a common laboratory contaminant, the artifact threshold is 2574 μ g/kg.

4.9.2 Water-to-Soil Conversion for VOCs

For VOCs, the formula for converting a water result to a soil result is not straightforward; the laboratory should be consulted before the convention used for organic extraction methods can be used to evaluate VOCs field blank results. In some cases, the raw data will show an "oncolumn" result reporting the concentration in the extract not converted to the final units used for the matrix of the samples. In these cases, the on-column results for field blanks can be multiplied by 5 (or 10) and compared directly to the on-column results reported for the associated field samples. It is more likely; however, that the laboratory software will show the raw data results already converted to the matrix units and this method of comparison will be usable only in a limited number of cases.

4.9.3 Water-to-Soil Conversion for Metals

For metals, the conversion equation is as follows:

$$C_S = (C_W \times V_F)/M_E$$

Where:

 C_s = the calculated equivalent solid concentration (in milligrams per kilogram [mg/kg])

 C_W = the reported aqueous concentration in $\mu g/L$

 V_F = The final volume of soil digestate extracts in liters (L)

 M_{E} = The nominal mass extracted for solid samples in grams (g) (use the mass of a solid method blank)

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Example: A rinse blank has a detected concentration of 5.3 μ g/L. The laboratory's preparation forms show that the final volume of soil extracts is 50 mL (= 0.05 L) and the soil method blank was extracted using 1.00 g. The rinse blank result is the equivalent of 0.265 μ g/g = 0.265 mg/kg, which leads to an artifact threshold of 1.325 mg/kg. Note that the laboratory may report an actual mass for the method blank that is not a "round" number. If it can be determined that that the nominal method blank mass is a round number like 1.00 g or 0.50 g, use that value even if an individual method blank may be slightly off (for example, 1.02 g or 0.49 g).

4.10 FIELD DUPLICATE PRECISION

The evaluation of field duplicate precision depends on the concentration of each target analyte detected in the duplicate pair relative to the LOQ. Concentrations can be considered "low-level" or "high-level." The QAPP will specify the criteria for making this determination, and this determination should be made for every detected analyte before any further duplicate evaluation. One of the most common criteria for determining if a pair of results is high-level is if both results are greater than 5 times the associated LOQ.

General rules for evaluating field duplicate results include the following elements in the sequential order they are presented:

- 1. Two nondetected results are considered to be in control.
- 2. Two results detected below the LOQ, or one result below the LOQ and one nondetected result are considered to be in control.
- 3. Two low level results or one low level-result and one high-level result are considered to be in control if the absolute difference of the two results is less than the value of the LOQ (in some cases, a criterion of less than 2 times the LOQ is used).
- 4. Two high-level results are considered to be in control if the RPD of the two results meets the RPD acceptance criterion listed in the QAPP.

4.11 SURROGATE RECOVERIES

As discussed in Section 3.2 of the SOP, the validator should verify that the surrogate control limits reported by the laboratory match those required in the project QAPP. Although some data validation conventions assign individual surrogate compounds to lists of target compounds, HGL discourages this practice and the preferred approach is to assume that all surrogate discrepancies are associated with all target analytes. An exception to this is the evaluation of SVOCs surrogate results. When evaluating surrogate recoveries for this method, the acid extractible fraction surrogates should be associated with the acid extractible fraction target compounds and the base/neutral extractible surrogates should be associated with the base/neutral extractible fraction target compounds.

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Surrogate recoveries that are above the acceptance limits are usually considered not to affect nondetected results. In cases of extremely high recoveries (approaching 200 percent or greater) the validator should consider whether an analytical system problem has occurred. If the cause for abnormally high recoveries is not noted in the case narrative, the validator should contact the laboratory and request an explanation for such anomalies. In some cases, such discrepancies can be traced to accidental double-spiking, and the recoveries will meet acceptance criteria when calculated using the actual spiked concentration. However, the validator should consider the qualification of nondetected results associated with unusually high recoveries if the underlying cause indicates a problem in the analytical system.

When extremely low surrogate recoveries occur (less than 10 percent), HGL's preferred protocol is to reject both detected and nondetected results; however, the qualification of such results should be evaluated against the requirements of the governing regulatory body.

Dilution of samples can affect surrogate recovery performance. Surrogate compounds should be added to a sample before any dilution steps. As a consequence, surrogate discrepancies can occur that are not caused by matrix or analytical effects but rather are caused by dilution effects. The validator should examine surrogate discrepancies in diluted analyses. In most cases, surrogate discrepancies reported in samples diluted 5 times or higher should be considered to be a dilution effect and qualification should not be applied to the affected sample results.

4.12 METHOD-SPECIFIC QC CHECKS

Method-specific QC elements include such checks as pH buffer checks, cyanide distillation standards, synthetic precipitation leaching procedure extraction blanks, and replicate precision for total organic carbon. If these checks are reported in a Level II data package, the validator should review these items. If the review guidelines are not included in the QAPP, the validator should consult with the project chemist to develop a review and qualification approach.

4.13 ANALYTE QUANTITATION

The validator should discuss any dilutions performed. In some cases, multiple analyses will be performed on a sample because of a required dilution or to verify results affected by a QC discrepancy. Some laboratories will report the entire analytical dataset for all analyses performed on a sample, while others will report only the "best" result for each analyte. If the laboratory reported multiple results for an analyte or set of analytes in a sample, the validator should select the best result for each analyte in each sample and indicate which result was chosen and why in the validation narrative. All results that are not selected for use are excluded from the dataset, and this is indicated by appending an X qualifier to the laboratory applied qualifiers.

Samples that are nominally solid samples may have very high percent moisture content. This is especially true of sediment samples that are very "soupy." Calculation of concentration on a

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dry weight basis for solid samples composed of less than 50 percent solids is complicated by the added nonhomogeneity of the samples.

5.0 LEVEL III QC ELEMENTS

The Level II validation guidelines above are applicable to QC elements that are common to many analytical methods. Level III validation guidelines address QC elements that are more specific to individual extraction and analytical principles.

5.1 GC/MS ORGANICS

Gas chromatography (GC)/mass spectrometer (MS) organics include analyses for VOCs and for SVOCs, most commonly by SW-846 methods 8260B and 8270C, respectively.

5.1.1 Instrument Tuning

SW-846 GC/MS methods require that the MS be tuned at the beginning of each 12-hour analytical sequence. MS tuning is a critical QC component and no analyses may proceed without an acceptable MS tuning. Each GC/MS method document prescribes the ions of interest and the required relative abundances. If MS tuning data show discrepancies and sample analyses proceeded without corrective action, the project chemist should be contacted immediately to resolve this issue.

In some cases, laboratories report tuning criteria for CLP analysis methods for SW-846 analyses. Although this approach is permissible, it is not in accordance with the QAPP. When the validator observes incorrect MS tuning criteria applied to tuning results, she or he should immediately contact the project chemist to determine if the affected results are usable and to initiate corrective action at the laboratory.

5.1.2 Instrument Initial Calibration

Most GC/MS analytes will be calibrated to a mean relative response factor (RRF), which quantitatively relates the concentration of each target analyte to the associated internal standard. There should be at least 5 calibration points for an initial calibration to a mean RRF to be valid. If the calibration relationship for a compound is linear or quadratic, a minimum of 6 and 7 points, respectively, is required.

5.1.2.1 Instrument Performance Criteria

For an initial calibration to be valid for GC/MS methods, system performance check compounds (SPCC) and calibration check compounds (CCC) are critical QC elements and must meet acceptance criteria, even if these method-specified compounds are not target analytes for the associated samples. One exception to this statement is if SVOCs analyses are only requested for base/neutral-extractable compounds or acid extractable compounds, only

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the SPCCs and CCCs associated with that fraction need be reported and evaluated. Each SPCC must meet minimum RRF requirements, even if an individual SPCC is calibrated to a linear or quadratic relationship. Each CCC must meet maximum percent relative standard deviation (%RSD) requirements, even if an individual SPCC is calibrated to a linear or quadratic relationship. Failure of these compounds to meet acceptance criteria can indicate instrumental problems such as dirty injector ports, carrier gas flow problems, or reactive sites on the chromatography column. Consequently, analyses performed in association with failed SPCCs and CCCs are potentially compromised by instrument performance.

If SPCC or CCC discrepancies are noted, this information must be referred to the HGL senior chemist and project manager for immediate follow-up with the laboratory. SPCC and CCC discrepancies are serious QC deficiencies and can potentially result in the rejection of all data produced in association with that initial calibration. The HGL senior chemist, the HGL project manager, and the laboratory project manager and QC manager will determine (1) if the associated results can be used, (2) the appropriate instrument maintenance and recalibration procedures, and (3) the notification measures to ensure that SPCC and CCC deficiencies are appropriately addressed at the laboratory as soon as they are noted by the analyst.

Note that an SPCC or a CCC that is also a target compound will be evaluated against both the SPCC or CCC acceptance criteria and against the target analyte criteria presented in Section 5.1.2.2 below. These two evaluations are independent of each other.

Example: VOCs CCC vinyl chloride is reported calibrated to a mean RRF with %RSD of 17.5 percent. The requirement for VOCs CCCs is that each have a %RSD of no greater than 30 percent. Vinyl chloride shows acceptable performance as a CCC; however, the target analyte criterion is for %RSD to be no greater than 15 percent. Vinyl chloride does not meet the acceptance criterion for target analytes. The effects, if any, of this discrepancy would be considered to affect vinyl chloride alone and not to be indicative of an instrument performance issue.

Laboratory initial calibration summary form formats will vary. If SPCCs are reported as calibrated to a linear or quadratic relationship, some laboratories' summary reporting forms may present the m1 term associated with the curve instead of the mean RRF. Other laboratories' summary forms may present both. If the summary forms do not include the mean RRF for one or more SPCCs, the validator should examine the associated continuing calibration verification forms; on occasion, the initial calibration mean RRF is reported there in addition to the continuing calibration RRF. The mean RRF also may be discussed in the case narrative if HGL has requested the laboratory to do so. If the mean RRF is not available in other locations in the data package, the data validator should contact the laboratory project manager and have this information transmitted.

As with SPCCs, laboratory summary forms may not present the CCC %RSDs for those CCCs calibrated to linear or quadratic relationships. This information is generally not presented elsewhere in the data package unless HGL has made arrangements with the project laboratory

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to present this information in the case narrative. Otherwise, the data validator should contact the laboratory project manager and have this information transmitted.

5.1.2.2 Target Analyte Performance Criteria

The linearity criterion for GC/MS initial calibration is %RSD no greater than 15 percent. The correlation (r^2) of linear or quadratic relationships should be no less than 0.990.

SW-846 methods do not have a requirement for minimum mean RRF for target analytes; however, some historical project QAPPs may include a requirement for all target analytes to show a mean RRF of no less than 0.050. This requirement comes from the requirements of the CLP Scope of Work and associated data validation protocols. The laboratory's summary forms may not present this information for target analytes calibrated to linear or quadratic relationships. If so, the validator should review the continuing calibration forms and case narrative to determine if this information is available from other sources, as described in Section 5.1.2.1 above.

5.1.3 Second Source Calibration Verification

A second source calibration verification standard should be analyzed immediately after the initial calibration is performed. The performance of each target analyte should be evaluated against the acceptance criteria presented in the QAPP. SPCC and CCC performance evaluation is not required for second source calibration verification standards.

5.1.4 Instrument Continuing Calibration

Continuing calibration standards must be analyzed immediately after an acceptable MS tuning has been performed. Continuing calibration standards are reviewed for SPCC, CCC, and target analyte performance in a manner similar to the evaluation performed for initial calibrations. SPCCs must meet method-specified continuing calibration RRF criteria and CCCs must meet method-specified percent difference (%D) criteria. Target analytes are evaluated against the target analyte criterion of no greater than 20 percent, and some QAPPs may also require that target compounds also meet minimum continuing calibration RRF criteria.

Note that some laboratories evaluate continuing calibration results with respect to the direction of the bias and consider nondetected sample results associated with a discrepancy biased high to be acceptable. HGL's preferred convention is to consider all continuing calibration discrepancies to affect detections and nondetections regardless of direction of bias.

5.1.5 Internal Standards

Internal standard compounds must be spiked into every sample, standard, and blank analyzed by GC/MS methods. Internal standards must meet the method area and retention time criteria

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for peak area and retention time. The peak area for each internal standard compound must be no less than 50 percent and no greater than 200 percent of the peak area for that internal standard compound in the midpoint standard in the associated initial calibration sequence. The retention time for each internal standard must be within 30 seconds of the retention time of the midpoint standard in the associated initial calibration sequence.

Discrepancies in internal standard performance are generally associated with the matrix characteristics of individual samples and are not usually indicative of an instrument issue. Internal standard discrepancies should always be associated with a corrective action by the laboratory, which will usually consist of re-extraction and reanalysis of the affected samples. The only exception is if the internal standards that exhibit discrepancies are not associated with any target analytes.

Each internal standard is associated with a specific set of analytes. When internal standards are out of control, only the associated target analytes are qualified in the affected sample. Many formats of initial calibration summary forms are organized to show the internal standard associations. If the internal standard associations are not shown on the initial calibration summary or other form, the validator should contact the laboratory to have the required information transmitted.

5.2 GC AND HPLC ORGANICS

GC and high performance liquid chromatography (HPLC) organics include analyses for pesticides (organochlorine and organophosphorus), PCBs, PAHs, explosives, herbicides, and petroleum products. GC and HPLC analyses use dual columns or dual detectors to identify target analytes. Some laboratories assign the same quantitative significance to both columns/detectors, while others specify a dedicated primary and secondary column/detector. If presented, the QC data for both the primary and secondary column/detector should be evaluated. In cases where instrument QC discrepancies affect one column/detector and not the other, some degree of interpretation by the validator is required to determine the effect on the associated samples.

5.2.1 Instrument Initial Calibration

As with GC/MS methods, initial calibrations must include at least five calibration points for calibration to response factor. Six calibration points are required for linear calibration and seven calibration data points are required for quadratic calibration. Initial calibration to response factor is required to meet the method-specific requirement, which is usually a %RSD no greater than 15 percent or 20 percent.

The analysis of PCBs only requires multipoint calibration for PCB-1016 and PCB-1260, with single point calibration for all other reported PCB congeners. PCBs are quantified using five characteristic peaks. The *mean* %RSD of the PCB-1016 peaks and the mean %RSD of the PCB-1260 peaks are compared to the acceptance criteria. Individual characteristic peaks may

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exceed the %RSD criterion so long as the mean %RSD for each congener is acceptable. Generally, discrepancies shown by PCB-1016 are considered to affect PCBs 1016, 1221, and 1232; and discrepancies shown by PCB-1260 are considered to affect PCBs 1242, 1248, 1254, and 1260. If PCBs other than 1016 or 1260 are identified in any associated sample, the laboratory should perform a multipoint calibration for all identified congeners and reanalyze the samples to quantify the detected congeners. These reanalyses should be accompanied by all other QC elements spiked with the specific detected PCBs and not with the representative PCB-1016/1260 mixture.

5.2.2 Second Source Calibration Verification

A second source calibration verification standard should be analyzed immediately after the initial calibration is performed. The performance of each target analyte should be evaluated against the acceptance criteria presented in the QAPP.

Because of the existence of multiple overlapping peaks in the spectrum of each individual PCB congener, PCBs second source calibration verifications are spiked with a mixture of PCB-1016 and PCB-1260. Generally, discrepancies shown by PCB-1016 are considered to affect PCBs 1016, 1221, and 1232; and discrepancies shown by PCB-1260 are considered to affect PCBs 1242, 1248, 1254, and 1260.

5.2.3 Instrument Continuing Calibration

GC and HPLC methods require a continuing calibration standard to be analyzed at the beginning of each analytical sequence, at regular intervals after a specified number of sample analyses (generally 10), and at the end of the end of the analytical sequence. Each continuing calibration standard is associated with all samples analyzed after the previous continuing calibration standard analysis and before the following continuing calibration standard analysis. Discrepancies in continuing calibration standard analyses will require evaluation of the affected analytes in the associated samples.

As a result of the existence of multiple overlapping peaks in the spectrum of each individual PCB congener, PCBs continuing calibration verification standards are spiked with a mixture of PCB-1016 and PCB-1260. Generally, discrepancies shown by PCB-1016 are considered to affect PCBs 1016, 1221, and 1232; and discrepancies shown by PCB-1260 are considered to affect PCBs 1242, 1248, 1254, and 1260.

Note that some laboratories evaluate continuing calibration results with respect to the direction of the bias and consider nondetected sample results associated with a discrepancy biased high to be acceptable. HGL's preferred convention is to consider all continuing calibration discrepancies to affect detections and nondetections regardless of direction of bias.

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5.2.4 Degradation Summary

Analysis for organochlorine pesticides requires that a dichlorodiphenyltrichloroethane (DDT) and endrin degradation standard be measured before samples are analyzed and at the beginning of each 12-hour shift. These compounds are easily degraded at the injection port. Generally, the acceptance criterion is that neither DDT nor endrin should have a breakdown of greater than 15 percent. Unacceptable DDT breakdown will cause the qualification of all associated DDT, dichlorodiphenyldichloroethene (DDE), and dichlorodiphenyldichloroethane (DDD) results. Unacceptable endrin breakdown will cause the qualification of all associated endrin, endrin aldehyde, and endrin ketone results. However, this test should be performed as a test of the inertness of the analytical system even when DDT and endrin are not target analytes for a given project, unless otherwise specified in the QAPP.

5.2.5 Retention Times

There are no standardized summary forms for reporting chromatographic retention times and each laboratory's forms will vary greatly in both format and content. In general, the validator should review all available retention time data. Retention time shifts, either in calibration standards or in sample results, must be accompanied by analyst documentation for the associated results to be accepted.

5.2.6 Confirmation

GC and HPLC methods require confirmation to differentiate target analytes from matrix interferences. Detected results are confirmed either by a second detector or by retention time on a second column that has different chemical properties than the primary column. Target analytes detected on one column/detector that are not confirmed are potentially interferences rather than a true detection. Such results should not be reported as detections by the laboratory unless the analyst and section leader provide documentation as to why the analytes should be considered detected in the absence of confirmation. Results that are detected and confirmed should have approximately the same quantitation on both columns/detectors; results that do not meet RPD criteria should be qualified as estimated.

Confirmation is not required for multicomponent analytes such as gasoline range organics, diesel range organics, PCBs, toxaphene, and technical chlordane. If confirmation data is provided, however, it should be evaluated in the same manner as for those analytes requiring confirmation.

5.3 METALS

Metals analyses are performed using SW-846 methods 6010C (inductively coupled plasma-atomic emission spectroscopy [ICP-AES]) and 6020A (inductively coupled plasma-mass spectrometry [ICP-MS]) for "full list" metals; cold vapor atomic absorption (CVAA) methods 7470A and 7471B for mercury in water and soil, respectively; and graphite furnace atomic

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absorption (GFAA) method 7010 for select metals that can be affected by spectral interferences that prevented definitive analysis by ICP-AES. Although improvements to ICP-AES and the emergence of ICP-MS as the metals method of choice have decreased the need for GFAA analysis, it is still used in some situations.

5.3.1 Instrument Tuning

Method 6020A uses MS to identify target elements; the MS must be tuned prior to use. Instrument tuning data is not always available on summary forms. If the required data is not available for review on summary forms, the data validator should contact the laboratory to request the required information. If the information is not available on summary forms, the raw data must be examined.

The QSM requires that tuning peaks show a resolution of no greater than 0.9 atomic mass units (amu) at 10 percent peak height. Some instrumental systems report the peak resolution at 5 percent of total peak height; this is more stringent than the QSM requirement and should not be considered a discrepancy provided that the resolution criterion of ≤ 0.9 amu is met.

5.3.2 Internal Standards

Method 6020A uses internal standards in the quantification of target elements. If an internal standard does not meet acceptance criteria and corrective action was not performed or was not successful, the target analytes associated with that internal standard should be qualified in the affected sample.

In some cases (especially with short analyte lists), there may be internal standards that do not meet acceptance limits but are not associated with target metals. Some laboratories also will choose a secondary internal standard to quantify a metal if the primary internal standard does not meet acceptance criteria.

5.3.3 Initial Multipoint Calibration

Initial multipoint calibration is required for CVAA and GFAA methods. It is not required for ICP-AES or ICP-MS analyses and there are QC elements described below that are intended to be performed instead of initial multipoint calibration; however, if a multipoint initial calibration is performed, it must meet the acceptance criteria in the QAPP. If the alternative QC checks are acceptable but the multipoint initial calibration was out of control, the associated results must be considered for qualification. The laboratory should not present such a situation as being in control.

5.3.4 Low-Level Calibration Verification

Low-level calibration verification standards are required under projects with QC requirements from the QSM. This QC check should be performed for ICP-AES and ICP-MS methods if an

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initial multipoint calibration is not performed. Note that the DoD QSM requires that this check meet control limits of 80 to 120 percent even though the method allows a window of 70 to 130 percent. If an initial multipoint calibration has been performed, this QC check is not required. If the results are presented, however, they should be in control.

Some laboratories also perform what is called a CRDL check standard. This CRDL check standard is generally spiked at 2 times the LOQ. If the low-level calibration verification standard does not meet acceptance criteria, the usual response is to qualify detections with concentrations up to 10 times the LOQ and nondetections. However, if a low-level calibration verification does not meet acceptance criteria and an associated CRDL check standard is performed and is in control, stability at 2 times the LOQ has been demonstrated and only detected results up to 2 times the LOQ and nondetections require qualification.

5.3.5 High-Level Calibration Verification

High-level calibration verification standards are used to determine the upper end of the working range of the instrument. If the high-level calibration verification standard does not meet acceptance criteria, the validator should determine if a multipoint initial calibration has been performed. If so, and the high point on the calibrated curve has a concentration below that of the high-level calibration verification standard, only results above the high point on the curve (adjusted for matrix as necessary) require qualification.

Detected results above the high-level calibration verification should be qualified unless the laboratory performed appropriate dilutions so that the effective concentration measured by the instrument is less than the high-level calibration verification standard concentration.

5.3.6 Initial and Continuing Calibration Verification

Most laboratories use initial calibration verification (ICV) standard analyses as a second source verification check. HGL's preferred convention is to associate ICV results with all sample results in an analytical sequence and to the associated continuing calibration verification standard (CCV) results only with sample results "bracketed" by a given CCV. A result is considered bracketed by a CCV if that CCV is the last CCV analyzed before that result was generated or is the first CCV analyzed after that result is generated.

Note that some laboratories evaluate ICV/CCV results with respect to the direction of the bias and consider nondetected sample results associated with a discrepancy biased high to be acceptable. HGL's preferred convention is to consider all continuing calibration discrepancies to affect detections and nondetections regardless of direction of bias.

5.3.7 Continuing Calibration Blanks

Continuing calibration blanks (CCBs), including initial calibration blanks (ICBs), are performed for inorganic methods. CCBs are evaluated like method blanks (Section 4.8.).

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HGL's preferred convention is to associate ICB results with all sample results in an analytical sequence and to associated CCB results only with sample results bracketed by a given CCB. A result is considered bracketed by a CCB if that CCB is the last CCB analyzed before that result was generated or is the first CCB analyzed after that result is generated.

CCBs are aqueous, but can be associated with both aqueous and solid matrix analyses. When determining the potential effect of CCB contamination on the associated solid matrix sample results, convert the CCB result to an equivalent soil concentration using the procedure presented for field blanks (Section 4.9.3).

The artifact threshold associated with field blank contamination is 5 times the concentration detected in the blank (10 times the concentration in the case of common laboratory contaminants). As with action levels associated with method blank contamination, both aqueous and solid-equivalent action levels should be adjusted on a sample-specific basis to account for sample-specific variables. In most cases, it will be readily apparent that a result is above or below an action level and in practice this sample-specific adjustment is necessary for a minority of comparisons.

5.3.8 Interference Check Sample Results

Interference check samples (ICSs) are analyzed in pairs. ICS A (ICSA) is a blank spiked with high concentrations of aluminum, calcium, iron, and magnesium. ICS AB (ICSAB) is spiked with the same levels of aluminum, calcium, iron, and magnesium as is the ICSA and also contains lower spiked levels of all elements of concern. The purpose of analyzing ICSAs is to determine if interelement correction factors from naturally occurring elements that are often present at high concentrations cause false positive or false negative results due to over- or under-correction. The purpose of analyzing ICSABs is to determine if interelement correction factors for all elements, including those that occur at high concentrations naturally, are being applied correctly and provide correct quantitation. Generally, QAPPs will require a single ICSA and ICSAB be analyzed before sample analyses as a minimum requirement; however, if the laboratory reports multiple ICSA/ICSAB results in an analytical sequence, the reviewer should evaluate the bracketing ICSA/ICSAB results both before and after the sample analyses and assign both sets equal significance.

ICSA discrepancies can be an indicator of problems with interelement correction. HGL has had experiences with false positive results ultimately traced to failure of the analytical system to take advantage of all mathematical tools available to correct for interferences. In cases where ICSA discrepancies are attributable to known contamination in the stock solution, this situation should be noted by the laboratory in the case narrative. In other cases, ICSA discrepancies can be attributed to instrument drift or system contamination. Indicators of this kind of issue will include positive or negative results in associated CCBs or method blanks. If ICSA discrepancies are potentially attributable to other sources, the reviewer should consider not qualifying the associated results or reducing the severity of qualification.

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Most data validation conventions consider ICSA results with absolute value greater than the LOQ to constitute a severe discrepancy. If severe ICSA discrepancies are noted, the data reviewer should contact the HGL senior chemist before rejecting the associated results. ICSAs often contain higher levels of interfering element concentrations than are present in environmental samples, and alternatives to rejection may be available. Note that ICSA results are reported in aqueous units. If an ICSA associated with soil sample analyses shows a severe discrepancy based on comparison to the aqueous LOQ, but the ICSA result is less than the soil LOQ when the units are converted, this should be narrated but should not be considered a severe discrepancy.

It is very rare for ICSAB results to fail to meet control criteria, and often this is an indication of a spiking error rather than a problem with the analytical sequence.

5.3.9 Serial Dilution Results

Serial dilutions are performed to verify that the sample matrix does not interfere with the quantification of associated results. Serial dilutions must be performed on a site sample on a preparation batch-specific basis. Note that in some cases, the laboratory will report serial dilution results from a different SDG as batch control without the client sample ID. When a batch control serial dilution is reported, the validator should use the laboratory sample ID to confirm whether the serial dilution is actually from a site sample reported in a different SDG or from a nonsite sample. If the serial dilution parent sample is a site sample, it will be considered applicable to associated results.

Serial dilution results are evaluated on an analyte-specific basis. Serial dilution results are only applicable if the parent sample concentration is greater than 50 times the DL (method 6010C), 100 times the DL (method 6020A), or 25 times the DL (methods 7470A/7471B).

Data qualification is not applied on the basis of serial dilution results alone for analysis by methods 6010C or 6020A. These methods require that corrective action in the form of a post-digestion spike (PDS) analysis be performed under one of these three circumstances: (1) serial dilution is not performed; (2) the serial dilution result for an analyte does not meet the acceptance criterion and the parent sample concentration meets the minimum DL requirement for that method or (3) the parent sample concentration for one or more reported analytes does not meet the minimum DL requirement for that method.

Mercury results are qualified based on serial dilution results alone. If a serial dilution was not performed or the serial dilution did not meet acceptance criteria and the mercury concentration in the parent sample was greater than 25 times the DL, the associated results should be qualified. If a serial dilution is performed and the mercury concentration in the parent sample is less than 25 times the DL, no qualification is required regardless of the serial dilution results.

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5.3.10 Post-Digestion Spike Recoveries

PDS results are used in conjunction with serial dilution results. If serial dilution results for methods 6010C or 6020A are nonconforming for one of the three reasons given in Section 5.3.8 above, PDS results are used to determine if qualification is required. If serial dilution results show discrepancies or serial dilution was not performed, the associated results are qualified if the PDS also shows a discrepancy for the affected elements. Generally, if serial dilution results for a specific element do not conform but PDS results are in control, no qualification is required. If the serial dilution is in control, it is not necessary to qualify based on PDS discrepancies alone.

PDS discrepancies in samples that have concentrations of the affected target analytes greater than 4 times the spiked concentration are not considered applicable. If the affected analytes showed a discrepancy in the serial dilution, then the results for these analytes should be qualified in the associated samples.

If the laboratory performed neither a serial dilution nor a PDS using a project sample, then matrix effects cannot be ruled out. The validator should review available MS/MSD data, site results reported from other data packages, and the case narrative and determine whether qualification is necessary.

5.3.11 Recovery Test Recoveries

GFAA methods use recovery tests to determine if the sample matrix has an effect on reported results. The method requires a recovery test to be performed on a representative sample in each preparation batch, but in practice, laboratories perform recovery tests on a sample-specific basis.

5.3.12 Method of Standard Addition Results

The method of standard additions (MSA) is associated with GFAA analyses; this procedure is rarely performed as virtually all laboratories perform sample-specific recovery tests rather than batch-specific recovery tests. If MSA results are reported in a data package, the data validator should consult with the HGL senior chemist.

5.4 GENERAL CHEMISTRY

General chemistry parameters include a wide variety of analytical parameters and methodologies, including colorimetry, ion chromatography, GC, and infrared spectrometry. Usually, these parameters are secondary data that are used to determine the potential for a site to undergo monitored natural attenuation or the progress of monitored natural attenuation. Often, these tests will only require a Level II data review; however, some parameters, such as cyanide, perchlorate, anions, or total organic carbon will, on occasion, require Level III validation.

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In many cases, the review of general chemistry QC parameters is similar to the review of the corresponding parameters for metals. Method-specific QC parameters should be discussed in the QAPP along with the acceptance criteria and qualification requirements. Some laboratories do not have summary forms for Level III QC elements and the raw data will need to be examined by the validator to evaluate performance.

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ATTACHMENT D Automated Data Review

1.0 INTRODUCTION

Automated data review (ADR) is a proprietary data validation software platform developed by Laboratory Data Consultants, Inc. (LDC) of Carlsbad, California. The ADR program identifies quality control (QC) issues by comparing QC results in the laboratory-generated electronic database deliverable (EDD) against a data library generated in accordance with the requirements of the project Quality Assurance Project Plan (QAPP). ADR is capable of streamlining the data validation process by identifying QC issues and providing a listing of preliminary data qualification to be applied to the associated results; the extent of chemist review post-ADR will depend on project-specific requirements and objectives and on the EDD-generating capabilities of the laboratory.

2.0 ADR USES AND LIMITATIONS

ADR can reduce the amount of time spent reviewing laboratory data reports by generating a comprehensive list of QC discrepancies in a data package and identifying the associated affected results. ADR can be the primary data validation tool used for a project, integrated with only minimal "sanity check" review by a staff chemist, or it can be used as a tool to support manual data validation, relieving the validator from the task of reviewing each page of the laboratory data report and documenting all observed QC discrepancies.

ADR is capable of supporting Level II validation (as defined in Attachment A) and most of the elements of Level III validation; however, few laboratories provide Level III data elements in their EDDs and in practice ADR is used to provide the equivalent of a Level II data review. As laboratory EDD capabilities expand, it is expected that ADR will be able to be used for more extensive review of Level III QC elements.

2.1 LEVEL II REVIEW LIMITATIONS

ADR is not capable of evaluating the information in several critical areas of Level II data review. In some cases, the QC element is not included in ADR. In other cases, ADR is capable of performing an initial check of a QC element against the performance criteria but is not capable of incorporating additional sample- or method-specific information that is used to modify the initial evaluation. Following ADR, the ADR result should be reviewed by a staff chemist to ensure that all qualification applied by ADR is appropriate based on additional information not able to be evaluated by ADR.

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2.1.1 Case Narrative

ADR also cannot review any issues identified in the case narrative that may not be reflected in the associated QC data results. The case narrative should be examined by a chemist to ensure that there are no additional issues that require corrective action, resolution, or qualification of the associated data.

2.1.2 Sample Delivery and Condition

ADR is capable of qualification based on sample temperature at receipt; however, it cannot evaluate other issues associated with sample delivery and condition, including broken bottles, misidentified samples, improper preservation, and bubbles greater than 5 millimeter noted in volatile organic compound (VOC) sample vials. The staff chemist should review the chain of custody, the laboratory sample chronicle, and sample receipt documentation to verify that the samples were delivered to the laboratory in good condition, and properly identified.

2.1.3 Holding Times

Holding time can be evaluated by ADR. However, the holding time calculated from the time of collection on the chain of custody to the time of preparation or analysis at the laboratory can differ from the true holding time. This can be due to time zone differences between the sample location and the laboratory or a switch to or from daylight savings time occurring between the time of sampling and the time of preparation or analysis. The staff chemist should review the holding time calculations and ensure that these differences are accounted for.

Additionally, some projects require that the field teams assign "dummy" sample times to field duplicate samples to obscure the parent sample identity. The staff chemist should ensure that holding times for field duplicate samples have been calculated using the actual collection time and not an arbitrary collection time entered by the field sampling team.

2.1.4 Surrogate Recoveries

Sample dilution can cause surrogate recovery discrepancies that are not associated with matrix interferences or analytical problems. When ADR identifies surrogate discrepancies in diluted samples, the staff chemist should review the affected data. Generally, data from sample analyses performed at a fivefold or greater dilution should not be qualified for surrogate discrepancies unless a matrix effect is noted to have affected the sample even when analyzed under dilution.

2.1.5 Matrix Spike/Matrix Spike Duplicate Recoveries

Matrix spike (MS)/matrix spike duplicate (MSD) recovery discrepancies are not considered to have significance if the native concentration of the affected analyte in the parent sample is more than four times the concentration resulting from the spike (see Section 4.7 of Attachment

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C). In some cases, the native concentration of one or more target analytes is so high that the MS/MSD will be analyzed under dilution. Discrepancies in diluted MS/MSDs are likely to be a result of dilution effects rather than matrix effects, as the majority of material in a diluted sample will consist of material not representative of the site (that is, it will be analyte-free laboratory water) and unlikely to contain interferences. In some cases, MS/MSDs are analyzed without dilution but with one or more spiked compounds quantitated above the calibrated range. Quantification of results above the calibrated range is inherently less reliable and MS/MSD discrepancies can be caused by quantification errors.

ADR does not take the "four times" rule, the effects of dilution, or the effects of results quantitated above the calibrated range into account when assigning qualifiers for MS/MSD discrepancies. The staff chemist should evaluate the MS/MSD percent recovery discrepancies identified by ADR and determine if these results are truly indicative of a matrix effect or are caused by other factors that eliminate the need for qualification of the associated results.

In some cases, the laboratory will report MS/MSD results from a different sample delivery group (SDG) as batch control; such batch control MS/MSDs are often presented without the client sample ID. When a batch control MS/MSD is reported, the staff chemist should use the laboratory sample ID to confirm whether the MS/MSD is actually from a site sample reported in a different SDG or from a nonsite sample. If the MS/MSD is from a site sample, it will be considered applicable to associated results and any data qualification selected by ADR will be considered applicable. If the MS/MSD cannot be associated with a site sample, the results should be noted but no qualification should be applied unless the underlying cause of the discrepancy is suspected to be a problem with the analytical system.

2.1.6 Matrix Spike/Matrix Spike Duplicate Precision

As described in Section 4.7 of Attachment C, some laboratories compare the concentrations detected in the MS and the MSD to calculate precision rather than comparing the percent recoveries. This convention can lead to the resulting relative percent differences (RPD) being an incorrect representation of the analyte-specific precision. If the expected concentration in the MS is different than the expected concentration in the MSD, calculation of the RPD using a direct comparison of the detected concentrations is not relevant. The staff chemist should verify that the RPDs reported for MS/MSD results are calculated using the percent recoveries or that the expected concentration in the MS is the same as in the MSD. If the RPDs are calculated using noncomparable results, the validator should contact the laboratory and request that the calculations be performed using percent recoveries. If this information cannot be produced by the laboratory, the staff chemist will have to perform these calculations.

2.1.7 Field and Laboratory Duplicate Precision

ADR evaluates the performance of field and laboratory duplicates based on the calculation of the RPD of the results for the parent sample and duplicate. However, ADR will not evaluate duplicate performance in light of the commonly used convention for "low-level" results,

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usually defined as results that are less than 5 times the quantitation limit. Under most data validation protocols, low-level results are evaluated by comparing the absolute difference between the parent and duplicate result to the associated quantitation limits (see Section 4.10 of Attachment C). If ADR is used without supplemental manual review, there is a potential for data to be over-qualified for field or laboratory duplicate discrepancies.

2.1.8 PCB Discrepancy Associations

As described in Sections 4.6 and 4.7 of Attachment C, laboratory control samples (LCS) and MS/MSDs for polychlorinated biphenyls (PCBs) analysis are spiked with only two representative PCB congeners. Discrepancies affecting PCB-1016 are also considered to affect results for PCBs 1221 and 1232, and discrepancies affecting PCB-1260 are also considered to affect results for PCBs 1242, 1248, and 1254. ADR is not able to extend the association of a QC issue reported for one compound to other compounds. If the validation protocol for a project requires qualification of additional PCB congeners when QC discrepancies are noted for PCB-1016 or PCB-1260, this situation will have to be addressed by the staff chemist.

2.1.9 Selection of Final Result

In cases where multiple analysis results are reported for a sample as a result of dilution or reanalysis, all analyses are reviewed by ADR. Based on the body of QC data, the staff chemist should select one definitive result for each analyte in each sample in accordance with Section 3.4 of Attachment C. All other results for that analyte in that sample should be denoted as superseded by applying an X qualifier to the qualifiers applied by ADR.

2.2 LEVEL III REVIEW LIMITATIONS

An EDD that supports the full range of data review items of which ADR is capable will enable the automated review of the following Level III data validation items:

- Initial calibration
- Initial calibration verification (second source verification)
- Continuing calibration verification
- Instrument tuning (gas chromatography [GC]/mass spectrometry [MS] methods only)

ADR cannot provide an evaluation of system performance check compounds (SPCC) and calibration check compounds (CCC) results in GC/MS initial and continuing calibration standards if these compounds were calibrated to a curve rather than to mean relative response factor. The evaluation of SPCC and CCC performance is a critical Level III QC element, and any affected data that does not undergo additional manual validation will not meet the definition of definitive data.

PCB calibration is performed using only two representative congeners: PCB-1016 and PCB-1260. Discrepancies in either of these two congeners are associated as described in Section 2.1

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above. To ensure that all associated results receive the appropriate qualification, manual review must be performed whenever ADR identifies a discrepancy in any PCB calibration result that would require qualification of data.

Level III data review elements that ADR cannot currently address include the following:

- Internal standards (GC/MS and inductively coupled plasma (ICP)/MS analyses);
- Instrument tuning (ICP/MS analyses);
- Dichlorodiphenyltrichloroethane (DDT)/endrin degradation standards (organochlorine pesticides);
- Retention times (GC and high performance liquid chromatography [HPLC] analyses);
- Second column or second detector confirmation (GC and HPLC methods);
- Initial and continuing calibration blanks (inorganics);
- Low- and high-level calibration verification standards (ICP metals);
- Interference check samples (ICP metals);
- Serial dilutions (ICP metals);
- Post-digestion spikes (ICP metals);
- Recovery tests (GFAA metals and mercury); and
- Method of standard additions results (metals).

Even when provided with a laboratory EDD that includes the most extensive list of data QC elements that are supported by ADR, a Level III data review cannot be completed using ADR alone. The listed QC elements must be manually reviewed to complete a Level III data validation on a laboratory data report.

3.0 ELECTRONIC QAPP AND DATA LIBRARY

All ADR functions require reference to the project-specific data library that is assembled into an electronic QAPP (eQAPP). It is critical that the eQAPP be prepared and the associated data library transmitted to the laboratory before project sampling activities. If the data library has not been constructed at the time of sample analysis, the required information may not be captured in the laboratory EDD, resulting in the need to regenerate EDDs that conform to the data library requirements or late EDD delivery, causing delays and potentially increased laboratory costs.

The eQAPP should encompass the sensitivity limits, control limits, validation protocols, qualification conventions, and qualifier priorities that have been established in the project

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QAPP. The data library requires the input from an HGL database manager, an HGL project chemist, and the laboratory database manager at a minimum. After the draft eQAPP has been prepared, all information contained in it must undergo a QC review against the requirements of the QAPP by an HGL chemist. Any discrepancies between the eQAPP and the QAPP must be resolved before the eQAPP can be used to support ADR.

3.1 SENSITIVITY LIMITS

There are two principal conventions for establishing sensitivity limits. Both are in common use and are described in Table C.1. ADR file formats are capable of supporting either sensitivity limit convention, as specified in the project QAPP.

3.2 CONTROL LIMITS

The method- and matrix-specific control limits listed in the QAPP should be incorporated into the eQAPP. Control limits can be differentiated by QC element (such as LCS/LCSDs and MS/MSDs).

3.3 VALIDATION PROTOCOLS

The project-specific validation protocols are entered into the eQAPP using the Qualification Scheme application of the ADR program. The Qualification Scheme for a project must match the procedures presented in the project QAPP. The Qualification Scheme allows for qualifiers to be assigned on the basis of whether each affected result is a detection or a nondetection. The Qualification Scheme also allows for discriminating between minor discrepancies and major discrepancies that require results to be rejected, i.e., several QC elements allow the entry of both an estimation limit and a rejection limit for that element.

3.4 QUALIFICATION CONVENTIONS

The Qualification Scheme includes the project-specific qualifiers that will be applied to analytical results either as a result of quantification (for example, results below the quantitation limit) or as a result of a QC discrepancy. The eQAPP can specify on a method-specific basis whether some QC elements, such as MS/MSD results, affect the parent sample only or all samples in the associated preparation batch.

3.5 QUALIFIER PRIORITY

ADR includes a Qualifier Hierarchy matrix that allows for the determination of the final qualifier applied to each data point. The Qualifier Hierarchy matrix only allows for the simultaneous evaluation of two qualifiers; if more than two qualifiers are potentially applicable to a sample result, ADR will evaluate only the two highest priority qualifiers as defined in the QAPP.

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4.0 ADR LABORATORY DELIVERABLES

The laboratory ADR-compatible EDD is divided into three files: (1) the Analytical Results Table (A1 File), (2) the Laboratory Instrument Table (A2 File), and (3) the Sample Analysis Table (A3 File). The A2 file is optional and contains the instrument QC elements that can be used to evaluate the specific Level III QC elements described in Section 2.2. The A1 and A3 files are required. The specifications for populating the fields in each of these files are available from LDC.

5.0 ADR PROCEDURES

At a minimum, each ADR EDD delivered by the laboratory will undergo a QC review upon receipt and QC sample associations will be added to the file. If additional manual review is required after the QC and association step, the procedures described in Sections 5.1 and 5.2 must be followed.

5.1 ADR FILE QC

On receipt from the laboratory, each set of EDD files should be reviewed to ensure that all required fields have been populated correctly and that all information is complete and correct. Following this QC check, the field QC sample results in the laboratory data package must be associated with the field sample results. This step includes associating trip blanks and equipment blanks with the corresponding field samples, and associating designated field duplicate samples and MS/MSDs with the corresponding parent samples.

5.2 SUPPLEMENTAL MANUAL REVIEW – LEVEL II

Manual chemist review of Level II QC elements should include the following elements, in accordance with the referenced guidance presented in Section 2.1 of Attachment D and the referenced sections of Attachment C:

- Case narrative (Section 4.1), including any associated sample discrepancy reports;
- Chain of custody (Section 4.2);
- Sample receipt and log-in forms (Section 4.3);
- Sample ID cross reference (Section 4.4);
- Association of PCB QC discrepancies with additional congeners (Sections 4.6 and 4.7);
- Evaluation of any MS/MSD results potentially not relevant to sample results (Section 4.7); and

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• Evaluation of any low-level field duplicate and laboratory duplicate comparisons (Section 4.10).

Any changes made to the ADR results based on manual review must be documented and undergo a peer review.

5.3 SUPPLEMENTAL MANUAL REVIEW – LEVEL III

Manual chemist review of Level III QC elements should include the all the elements listed in Section 5.2 above, as well as the following elements, in accordance with the referenced guidance presented in Attachment C:

- GC/MS initial calibration and continuing calibration results for SPCCs and CCCs calibrated to curves (Section 5.1.2.1 and 5.1.4);
- Internal standards for GC/MS (Section 5.1.5) and ICP/MS (Section 5.3.2);
- Instrument tuning for ICP/MS (Section 5.3.1);
- DDT/endrin degradation standards for organochlorine pesticides (Section 5.2.4);
- Association of PCB calibration discrepancies with additional congeners (Sections 5.2.1, 5.2.2, and 5.2.3);
- Retention times for GC and HPLC analyses (Section 5.2.5);
- Second column or second detector confirmation for GC and HPLC methods (Section 5.2.6);
- Initial and continuing calibration blanks for inorganics (Section 4.12);
- Low- and high-level calibration verification standards for ICP metals (Sections 5.3.4 and 5.3.5);
- Interference check samples for ICP metals (Section 5.3.7);
- Serial dilutions for ICP metals (Section 5.3.8);
- Post-digestion spikes for ICP metals (Section 5.3.9);
- Recovery tests for GFAA metals and mercury (Section 5.3.10); and
- Method of standard additions results for metals (Section 5.3.11).

Any changes made to the ADR results based on manual review must be documented and undergo a peer review.

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ATTACHMENT E Data Qualification Reason Codes

QC Element Code Definition Ambient Blank ABH Ambient blank result ≥ LOQ Ambient Blank ABHB Result is judged to be biased high based on associated ambient blank result Ambient Blank ABL Ambient blank result < LOQ Analyte Quantitation ACR Result above the upper end of the calibrated range Analyte Quantitation EXC Result excluded; another data point for this analyte was selected for use (use with X-qualified results) Analyte Quantitation RTW Target analyte outside retention time window Analyte Quantitation PSL Solid matrix sample with percent solids less than 10% Analyte Quantitation TR Result between the DL and LOQ Calibration Blank CBH Initial or continuing calibration blank result ≥ LOQ Calibration Blank CBH Result between the DL and LOQ Calibration Blank CBL Initial or continuing calibration blank result < LOQ Calibration Blank CBL Initial or continuing calibration blank result with absolute value < LOQ Calibration Blank CBN Negative initial or continuing calibration blank result with absolute value < LOQ Continuing Calibrati		Reason	
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Initial Calibration ICLS Initial calibration low-level standard >LOQ	Initial Calibration	ICLS	

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ATTACHMENT E (continued) Data Qualification Reason Codes

	Reason	
QC Element	Code	Definition
Initial Calibration	ICR2	Initial calibration r ² below acceptance criterion
Initial Calibration	ICRD	Initial calibration %RSD above acceptance criterion
Initial Calibration	ICRX	Initial calibration %RSD above acceptance criterion, extreme
		discrepancy
Initial Calibration	IRFL	Initial calibration RRF below acceptance criterion
Initial Calibration	ISPC	System performance check compound did not meet minimum mean
		RRF criterion in initial calibration
Initial Calibration	LQSH	LOQ check standard above acceptance criteria
Initial Calibration	LQSL	LOQ check standard below acceptance criteria
Initial Calibration	SSVD	Second-source standard did not meet %D criterion
Interference Check	ICAH	Non-spiked concentration above acceptance criterion in ICSA
Standard		•
Interference Check	ICAN	Negative concentration with absolute value above acceptance criterion
Standard		in ICSA
Interference Check	ICHX	Non-spiked concentration above acceptance criterion in ICSA,
Standard		extreme discrepancy
Interference Check	ICNX	Negative concentration with absolute value above acceptance criterion
Standard		in ICSA, extreme discrepancy
Interference Check	ICSH	ICSA or ICSAB spiked analyte with high %R
Standard		
Interference Check	ICSL	ICSA or ICSAB spiked analyte with low %R
Standard		
Internal Standards	IRH	Internal standard peak area above upper limit
Internal Standards	IRL	Internal standard peak area below lower limit
Internal Standards	IRLX	Internal standard peak area below lower limit, extreme discrepancy
Internal Standards	ISRT	Internal standard retention time outside window
Laboratory Control Sample	LCLX	LCS and/or LCSD %R below acceptance criterion, extreme
		discrepancy
Laboratory Control Sample	LCSH	LCS and/or LCSD %R above acceptance criterion
Laboratory Control Sample	LCSL	LCS and/or LCSD %R below acceptance criterion
Laboratory Control Sample	LCSP	LCS/LCSD RPD above acceptance criterion
Laboratory Duplicate	LDPA	Laboratory duplicate results did not meet absolute difference criterion
Laboratory Duplicate	LDPR	Laboratory duplicate results did not meet RPD criterion
Method Blank	MBH	Method blank result ≥ LOQ
Method Blank	MBHB	Result is judged to be biased high based on associated method blank
		result
Method Blank	MBL	Method blank result < LOQ
Matrix Spike	MSH	MS and/or MSD %R above acceptance criterion
Matrix Spike	MSL	MS and/or MSD %R below acceptance criterion
Matrix Spike	MSLX	MS and/or MSD %R below acceptance criterion, extreme discrepancy
Matrix Spike	MSP	MS/MSD RPD above acceptance criterion
Post-Digestion Spike	PDH	Post-digestion spike recovery high
Post-Digestion Spike	PDL	Post-digestion spike recovery low

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ATTACHMENT E (continued) Data Qualification Reason Codes

	Reason	
QC Element	Code	Definition
Post-Digestion Spike	PDLX	Post-digestion spike recovery low, extreme discrepancy
Post-Digestion Spike	PDN	Post-digestion spike not performed or not applicable and serial dilution result not performed or not applicable
Sample Delivery and Condition	BUB	Bubbles > 5 mm in VOCs vial
Sample Delivery and Condition	DAM	Sample container damaged
Sample Delivery and Condition	PRE	Sample not properly preserved
Sample Delivery and Condition	TEMP	Sample received at elevated temperature
Sample Delivery and Condition	TMPX	Sample received at elevated temperature, extreme discrepancy
Serial Dilution	SDIL	Serial dilution did not meet %D criterion
Serial Dilution	SDN	Serial dilution not performed
Surrogate	SS10	Surrogate %R low and <10%
Surrogate	SSH	Surrogate %R high
Surrogate	SSL	Surrogate $\%$ R low and $\ge 10\%$
Surrogate	SSN	Surrogate compound not spiked into sample
Trip Blank	TBH	Trip blank result \geq LOQ
Trip Blank	TBL	Trip blank result <loq< td=""></loq<>
Validator Judgment	VJ	Validator judgment (see validation narrative)

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ATTACHMENT F Review of Subcontracted Data Validation Reports

1.0 INTRODUCTION

The goal of subcontracted data validation is to generate a validated project dataset that is qualified in accordance with Quality Assurance Project Plan (QAPP) requirements and ready for HydroGeoLogic, Inc. (HGL) to upload into the project database, and to do so at a cost savings to HGL's projects. Subcontracted data validation will be performed in accordance with the individual firm's internal procedures and policies; however, the overall procedure must include prereview, validation by qualified personnel, and peer or senior review of all data validation reports before delivery to HGL. All validation should be performed in accordance with the project QAPP and the scope of work provided by HGL.

2.0 DELIVERABLES

2.1 SUBCONTRACTED DATA VALIDATOR

Subcontracted data validators will deliver data validation reports to HGL. These reports may be in the validation firm's internally derived format; however, HGL prefers that an individual report be prepared for each sample delivery group (SDG) and analytical method within that SDG (although "bundling" methods for metals and wet chemistry parameters is acceptable, in the same fashion as HGL's internally produced data validation reports). Each report should include a summary of every quality control (QC) element evaluated by the data validator, an identification of discrepancies, the qualification required by this discrepancy, and an identification of the associated samples. Subcontracted data validation reports are required to include a summary of all qualified data. This summary can be provided as a table of qualified results, as a listing of qualifiers assigned by QC element, or as copies of data reporting forms with validation qualifiers applied by hand.

In most cases, the subcontracted validator will also be responsible for providing qualified data electronically in a format that allows upload into HGL's project database (see Section 6.0 of the standard operating procedure [SOP]), usually in the form of an Excel file. The validation firm will be responsible for all data entry, data entry QC, and removal of any residual laboratory-applied flags before delivery to HGL.

2.2 HGL REVIEWER

The HGL reviewer should prepare a review report to document the findings of the review of each subcontracted data validation report. This review should include a discussion of all discrepancies noted, any followup communications with the data validator or the laboratory, and any changes to the final data qualifiers assigned by the laboratory. Each review report should be transmitted to the project manager, and project managers are responsible for

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ensuring that any HGL modifications to data qualifiers are correctly incorporated into the project database. An example of an HGL data validation review report is presented as Attachment F.1.

3.0 INITIAL HGL REVIEW

The initial data validation reports provided by the contractor should be reviewed in-depth by an HGL senior chemist as soon as possible to provide the data validator with timely feedback to guide ongoing validation efforts. Promptly alerting the data validators to these differences allows for data validator to issue correct reports rather than reissuing revised reports. Performing and in-depth review will assist in identifying areas where the data validation contractor's interpretation of QC elements differs from the requirements of the QAPP.

This review should mimic HGL's peer review of an internally generated data validation report (see Section 3.4 of the SOP), including a re-examination of the laboratory data package to verify that no QC discrepancies have been overlooked by the validator. The most common cause for a QC element being overlooked or misinterpreted by the data validator is unfamiliarity with the specific requirements of the project QAPP, which should supersede any corporate validation conventions in place at the validation firm.

4.0 GENERAL HGL REVIEW GUIDELINES

The following are the general guidelines for reviewing data validation reports from subcontracted validators.

4.1 REPORT DETAIL

When conducting data validation, HGL's practice is to identify and discuss all QC discrepancies associated with an analytical fraction, whether those QC discrepancies cause data to be qualified. Data validation subcontractors and individual validators vary in the amount of detail that is provided in the report narrative, especially if no corresponding results require qualification. The HGL reviewer should be alert to cases where the validator has indicated no discrepancies for a QC element when, in fact, there were discrepancies but no qualification is required or no project sample results are associated with that specific discrepancy.

4.2 APPLICATION OF FINAL QUALIFIERS

In all cases, the final qualifier applied by the data validator must be an allowable project qualifier. When more than one qualifier is applicable to a result, the final qualifier must have been assigned in accordance with the priority of qualifiers presented in the QAPP. Common errors are the overuse of the M qualifier because that qualifier has a much higher priority in the Air Force Center for Engineering and the Environment QAPP than in HGL's qualification conventions. M should never be applied to nondetected results or to results already qualified F (or J) as a result of being below the associated limit of quantitation (LOQ).

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The HGL reviewer should examine the qualified electronic file to ensure that all the validator-applied qualifiers are allowable under the project QAPP and that there are no changes to laboratory qualifiers that do not make sense. For instance, if a laboratory qualifier is U and the final qualifier is B, the HGL reviewer should suspect that the B qualifier is in error and determine what the correct final qualifier is applied.

5.0 REVIEW OF LEVEL II DATA VALIDATION ELEMENTS

The HGL reviewer should examine the following elements of each data validation report. The common discrepancies associated with each QC element are also discussed in the following subsections.

5.1 SAMPLE RECEIPT AND DELIVERY

The HGL reviewer should review the validation report and verify that any qualification is performed in accordance with the QAPP.

5.2 HOLDING TIMES

The holding times for preparation and analysis for each analytical method should be presented in the project QAPP. It is a common convention to evaluate holding times expressed in "days" on the basis of expired days; however, HGL's general convention is to measure holding times using both date and time; this convention should be included both in HGL's laboratory statement of work, the data validation statement of work and project QAPP.

Example: An aqueous semivolatile organic compound (SVOC) sample collected at 10:00 a.m. on May 1 would have a preparation holding time of 7 days, which would expire at 10:00 a.m. on May 8.

The validator should have used HGL's convention for evaluating holding times or provide justification (such as nominal exceedance) for not qualifying results that are associated with holding time exceedances. The validator should have taken into account any time zone differences and daylight savings time changes when evaluating holding time. Also, some sampling teams assign a "dummy" sample collection time (such as "1200") to field duplicate samples. Before qualifying field duplicate sample results for a holding time exceedance of less than a day, the data validator should have verified the actual sample collection time with the field team.

5.3 LCS/LCSD RECOVERIES AND PRECISION

Laboratory control sample (LCS) (and laboratory control sample duplicate [LCSD]) recoveries greater than the control limits should not cause qualification of nondetected results unless there is a gross exceedance that is evidence of a problem with the analytical system.

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LCS/LCSD relative percent difference (RPD) exceedances should not cause qualification of non-detected results.

Discrepancies shown by polychlorinated biphenyl (PCB)-1016 should be considered to affect PCBs 1016, 1221, and 1232; and discrepancies shown by PCB-1260 should be considered to affect PCBs 1242, 1248, 1254, and 1260. The validator should have taken this situation into account when applying qualifiers.

Some QAPP data validation protocols establish a two-tiered approach for evaluating LCSs; generally, low recoveries that are above the marginal exceedance threshold will cause associated nondetections

5.4 MS/MSD RECOVERIES AND PRECISION

The issues applying to LCS (and LCSD) performance also apply to matrix spike (MS)/matrix spike duplicates (MSDs). There are additional issues that affect the evaluation of MS/MSDs.

The association of MS/MSD results to project samples varies by method and by project. Ensure that any identified MS/MSD discrepancies are associated correctly.

Ensure that no qualification of project samples is performed based on discrepancies found in nonsite samples unless the validator has provided an appropriate rationale.

Ensure that no qualification has been performed based on MS/MSD discrepancies identified for analytes that are present in the parent sample at greater than 4 times the spiked concentration.

Ensure that project samples from other SDGs that were reported as batch control MS/MSDs were properly identified as project samples and used to qualify project data.

Verify that the RPDs reported for MS/MSD results are calculated using the percent recoveries or that the expected concentration in the MS is the same as in the MSD. If the RPDs are calculated using non-comparable results (different spiked concentrations in the MS and MSD), the validator should have noted this discrepancy.

5.5 METHOD BLANKS

The evaluation of laboratory blank results is one of the few QC elements where the results can meet acceptance requirements but the associated results will still be qualified. HGL often sets acceptance criteria for laboratory blanks as "no contamination found >2x detection limit (DL)" or "no contamination found >½ LOQ." These acceptance criteria are the thresholds above which the laboratory should take corrective action and evaluate the need to reanalyze any affected samples. However, HGL's convention is that any contamination detected in laboratory blanks above the associated DL must be used to establish an artifact threshold and

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qualify any associated results below that threshold. This qualification must be applied whether the associated blank result is above the acceptance criterion or below it.

This division between acceptance criteria and qualification criteria is a common source of error in subcontracted evaluation of laboratory blanks. The HGL review must ensure that the validator has evaluated all blank results above the DL and applied qualification in accordance with the validation conventions.

5.6 FIELD BLANKS

Field blanks are evaluated in a similar manner as method blanks (Section 5.5). Two main differences are (1) the artifact threshold calculated from concentrations in field blanks is *not* adjusted for sample-specific factors; and (2) most field blanks are aqueous and conversion to equivalent solid units is not straightforward for some analytical methods.

Ensure that the data validator correctly calculated the artifact threshold and made any corrections for conversion from water to soil units.

5.7 FIELD DUPLICATE PRECISION

Ensure that the appropriate criterion, absolute difference for low-level results of RPD for high-level results, was used to evaluate each set of duplicate results.

The association of field duplicate results to project samples beyond the parent sample varies by method and by project. Ensure that any identified field duplicate discrepancies are associated correctly.

5.8 SURROGATE RECOVERIES

The HGL reviewer should examine any results qualified as a result of surrogate discrepancies noted in diluted samples. Generally, qualification should not be applied for surrogate discrepancies if the sample dilution factor was 5 times or greater.

5.9 METHOD-SPECIFIC QC CHECKS

Method-specific QC elements include such checks as pH buffer checks, cyanide distillation standards, synthetic precipitation leaching procedure (SPLP) extraction blanks, and replicate precision for total organic carbon. If these checks are reported in a Level II data package, the validator should review these items. If the review guidelines are not included in the QAPP, the validator should consult with the project chemist to develop a review and qualification approach.

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6.0 REVIEW OF LEVEL III DATA VALIDATION ELEMENTS

Level II data QC elements are fairly generic; Level III QC elements are specific to individual analytical methods.

6.1 GC/MS ORGANICS

Gas chromatography (GC)/mass spectrometry (MS) organics include analyses for volatile organic compounds (VOC) and for SVOCs, most commonly by SW-846 methods 8260B and 8270C, respectively.

6.1.1 Instrument Tuning

It is rare for a laboratory data package to include mass spectrometer tuning discrepancies. Data validation reports for this QC element will rarely include more than a statement that tuning frequencies and results were acceptable.

6.1.2 Instrument Initial Calibration

A common source of error in subcontracted data validation reports is the confusion between instrument performance criteria and target compound performance criteria in the evaluation of initial calibration data. Subcontracted data validation reports should note that the following QC elements were reviewed, along with any noted discrepancies:

- System performance check compounds (SPCCs) evaluated against analyte-specific mean relative response factor (RRF)
- Calibration check compound (CCCs) evaluated against percent relative standard deviation (%RSD) of 30 percent
- Target analytes (including CCCs that are also target analytes) evaluated against %RSD of 15 percent or r² of 0.990

The failure of an SPCC or CCC to meet the SPCC- or CCC-specific criteria constitutes a failure of the entire calibration; whereas, the failure of a target compound to meet the linearity criterion constitutes a failure for only that target compound. In some cases, a CCC can pass the CCC criterion but fail the target analyte criterion.

Example: VOCs CCC vinyl chloride is reported calibrated to a mean RRF with %RSD of 17.5 percent. The requirement for VOCs CCCs is that each has a %RSD of no greater than 30 percent. Vinyl chloride shows acceptable performance as a CCC; however, the target analyte criterion is for %RSD to be no greater than 15 percent. Vinyl chloride does not meet the acceptance criterion for target analytes. The effects, if any, of this discrepancy would be considered to affect vinyl chloride alone and not to be indicative of an instrument performance issue.

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Some QAPPs include a requirement that target analytes also be evaluated against mean RRF requirements. This should only be done if included as a QAPP requirement; if the data validator has qualified data based on target compound mean RRF when not required by the QAPP, the data validation reports will be required to be revised to remove this extraneous qualification.

6.1.3 Second Source Calibration Verification

A second source calibration verification standard should be analyzed immediately after the initial calibration is performed. The performance of each target analyte should be evaluated against the acceptance criteria presented in the QAPP. SPCC and CCC performance evaluation is not required for second source calibration verification standards.

6.1.4 Instrument Continuing Calibration

The data validator should have evaluated continuing calibration standards for SPCC, CCC, and target analyte performance in a manner similar to the evaluation performed for initial calibrations. The data validation report should note that the SPCCs met method-specified continuing calibration RRF criteria and CCCs met method-specified percent difference (%D) criteria. Target analytes are evaluated against the target analyte criterion of no greater than 20 percent. Some QAPPs may also require that target compounds also meet minimum continuing calibration RRF criteria; if the QAPP does not require the evaluation of target compound RRFs, the data validation report should not use this QC element to assign qualifiers to target analyte data.

Note that some laboratories evaluate continuing calibration results with respect to the direction of the bias and consider nondetected sample results associated with a discrepancy biased high to be acceptable. HGL's preferred convention is to consider all continuing calibration discrepancies to affect detections and nondetections regardless of direction of bias. The data validation report should not use the direction of bias when evaluating continuing calibration results.

6.1.5 Internal Standards

Internal standard compounds must be spiked into every sample, standard, and blank analyzed by GC/MS methods. Internal standards must meet the method area and retention time criteria for peak area and retention time. The peak area for each internal standard compound must be no less than 50 percent and no greater than 200 percent of the peak area for that internal standard compound in the midpoint standard in the associated initial calibration sequence. The retention time for each internal standard must be within 30 seconds of the retention time of the midpoint standard in the associated initial calibration sequence.

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Note that some QAPPs have been written with the internal standard requirements keyed to the associated continuing calibration standard instead of the midpoint standard of the initial calibration sequence. The QAPP requirements should be used when validating data.

6.2 GC AND HPLC ORGANICS

GC and high performance liquid chromatography (HPLC) organics include analyses for pesticides (organochlorine and organophosphorus), PCBs, polynuclear aromatic hydrocarbons (PAHs), explosives, herbicides, and petroleum products. GC and HPLC analyses use dual columns or dual detectors to identify target analytes. Some laboratories assign the same quantitative significance to both columns/detectors, while others specify a dedicated primary and secondary column/detector. If presented, the QC data for both the primary and secondary column/detector should have been evaluated. In cases where instrument QC discrepancies affect one column/detector and not the other, some degree of interpretation by the validator is required to determine the effect on the associated samples.

6.2.1 Instrument Initial Calibration

The interpretation of GC initial calibration is generally straightforward. If any discrepancies are identified in the initial calibrations associated with PCBs analyses, the HGL reviewer should ensure that the validator considered discrepancies shown by PCB-1016 to affect PCBs 1016, 1221, and 1232; and considered discrepancies shown by PCB-1260 to affect PCBs 1242, 1248, 1254, and 1260.

6.2.2 Second Source Calibration Verification

A second source calibration verification standard should be analyzed immediately after the initial calibration is performed. The performance of each target analyte should be evaluated against the acceptance criteria presented in the QAPP. If any discrepancies are identified in the second source calibration verifications associated with PCBs analyses, the HGL reviewer should ensure that the validator considered discrepancies shown by PCB-1016 to affect PCBs 1016, 1221, and 1232; and considered discrepancies shown by PCB-1260 to affect PCBs 1242, 1248, 1254, and 1260.

6.2.3 Instrument Continuing Calibration

If any discrepancies are identified in the continuing calibration verifications associated with PCBs analyses, the HGL reviewer should ensure that the validator considered discrepancies shown by PCB-1016 to affect PCBs 1016, 1221, and 1232; and considered discrepancies shown by PCB-1260 to affect PCBs 1242, 1248, 1254, and 1260.

Note that some laboratories evaluate continuing calibration results with respect to the direction of the bias and consider non-detected sample results associated with a discrepancy biased high to be acceptable. HGL's preferred convention is to consider all continuing calibration

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discrepancies to affect detections and non-detections regardless of direction of bias. The data validation report should not use the direction of bias when evaluating continuing calibration results.

6.2.4 Degradation Summary

The evaluation of this QC element is straightforward and should not be a source of error in the validation report.

6.2.5 Retention Times

Verify that retention time shifts were evaluated in the data validation report.

6.2.6 Confirmation

Verify that confirmation was evaluated and that confirmed results were qualified if confirmation agreement criteria were not met.

6.3 METALS

Metals analyses often contain discrepancies between the validation criteria applied by the validator and the QAPP criteria. The HGL reviewer should be especially alert to errors in evaluating continuing calibration blanks (CCB) (Section 6.3.7), interference check samples (ICS) (Section 6.3.8) and the interaction of serial dilution results (Section 6.3.9) and post-digestion spike (PDS) results (Section 6.3.10).

6.3.1 Instrument Tuning

Instrument tuning data is not always available on summary forms. Verify that the validators were able to evaluate instrument tuning data, including mass windows, peak widths, and %RSD of scans.

6.3.2 Internal Standards

Verify that the validators reviewed internal standard results. In some cases (especially with short analyte lists), there may be internal standards that do not meet acceptance limits but are not associated with target metals. Some laboratories will also choose a secondary internal standard to quantify a metal if the primary internal standard does not meet acceptance criteria.

6.3.3 Initial Multipoint Calibration

Initial multipoint calibration is required for cold vapor atomic absorption (CVAA) and graphite furnace atomic absorption (GFAA) methods. It is not required for inductively coupled plasma (ICP) atomic emission spectroscopy (AES) or ICP-MS analyses and there are QC elements described below that are intended to be performed instead of initial multipoint calibration;

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however, if a multipoint initial calibration is performed, it must meet the acceptance criteria in the QAPP. If the alternative QC checks are acceptable but the multipoint initial calibration was out of control, the associated results should have been qualified by the validator.

6.3.4 Low-Level Calibration Verification

The integration of the results for initial calibration, low-level calibration standards, and contract required detection limit (CRDL) standards is a common source of validator error. The HGL validation reviewer should ensure that the validator understands how to evaluate these three QC elements in totality and apply the correct final qualifier to any results affected by discrepancies associated with the initial calibration QC checks.

6.3.5 High-Level Calibration Verification

Verify that the validator evaluated high-level calibration standards and qualified any results reported from above the calibrated range.

6.3.6 Initial and Continuing Calibration Verification

Most laboratories use initial calibration verification standard (ICV) analyses as a second source verification check. HGL's preferred convention is to associate ICV results with all sample results in an analytical sequence and to associate continuing calibration verification standard (CCV) results only with sample results "bracketed" by a given CCV. A result is considered bracketed by a CCV if that CCV is the last CCV analyzed before that result was generated or is the first CCV analyzed after that result is generated.

Note that some laboratories evaluate ICV/CCV results with respect to the direction of the bias and consider nondetected sample results associated with a discrepancy biased high to be acceptable. HGL's preferred convention is to consider all continuing calibration discrepancies to affect detections and non-detections regardless of direction of bias.

The HGL validation reviewer should ensure that the data validator correctly identified ICV/CCV results that did not meet acceptance criteria and that any discrepancies were associated in accordance with the QAPP conventions. The data validator should not have taken the direction of any ICV/CCV discrepancies into account when determining the qualification of the associated sample results.

6.3.7 Continuing Calibration Blanks

CCBs present the same common source of error as do method blanks: the confusion caused by the qualification criteria differing from acceptance criteria (see Section 5.5). The HGL reviewer should ensure that all CCB contamination above the DL was evaluated for the potential effect on associated sample results, not just the CCB contamination that was present above the acceptance criteria.

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CCBs are always aqueous; the concentrations should be converted to the equivalent soil concentration when comparing the blank results to the concentrations found in any associated soil samples. The HGL reviewer should verify that the appropriate conversion was made by the validator.

HGL's preferred convention is to associate ICB results with all sample results in an analytical sequence and to associate CCB results only with sample results bracketed by a given CCB. A result is considered bracketed by a CCB if that CCB is the last CCB analyzed before that result was generated or is the first CCB analyzed after that result is generated. The HGL reviewer should verify that the association conventions used by the data validator are those in the QAPP.

The HGL validation reviewer should ensure that the data validator correctly identified ICB/CCB results that did not meet acceptance criteria and that any discrepancies were associated in accordance with the QAPP conventions. The HGL reviewer should also verify that any blank contamination with concentrations or absolute values of concentrations greater than the acceptance levels were noted by the validator with a discussion of any laboratory corrective action.

6.3.8 Interference Check Sample Results

The evaluation of ICS data is another common source of error in data validation reports. One of the primary reasons for this is that laboratory data summary reporting forms generally provide inadequate information for the data validator to be able to evaluate the results that are presented. The HGL reviewer should evaluate whether the data validator evaluated ICS A (ICSA) results in accordance with the QAPP and applied the correct qualifiers. Common errors are: failure to evaluate ICSA results at all (some firms consider this a Level IV item); failure to identify severe discrepancies (results greater than the LOQ or converted water-to-soil LOQ); and failure to interpret discrepancies and apply qualification in accordance with the QAPP.

The evaluation of ICS AB results is generally straightforward, and this QC element rarely shows discrepancies.

6.3.9 Serial Dilution Results

The interpretation of serial dilution results and the integration of serial dilution results with PDS results (Section 6.3.10) are a common source of error. Often, subcontracted data validators overlook that both serial dilutions and PDSs should be performed on a project sample in each preparation batch. Subcontracted data validators will often overlook that if serial dilution results are not applicable as a result of parent sample concentrations being below the threshold, that PDS results should be evaluated to demonstrate that there are no matrix effects.

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SOP Category: Data Quality

Revision No.: 00 Date: November 2012

The HGL reviewer should evaluate the validation narrative and verify that serial dilutions and PDSs were evaluated in accordance with QAPP criteria and in the correct order.

6.3.10 Post-Digestion Spike Recoveries

Verify that PDS discrepancies in samples that have concentrations of the affected target analytes greater than 4 times the spiked concentration are not considered applicable. If the affected analytes showed a discrepancy in the serial dilution, then the results for these analytes should have been qualified in the associated samples.

If the laboratory performed neither a serial dilution nor a PDS using a project sample, then matrix effects cannot be ruled out. The validator should have reviewed available MS/MSD data, site results reported from other data packages, and the case narrative and determine whether qualification is necessary.

6.3.11 Recovery Test Recoveries

GFAA methods use recovery tests to determine if the sample matrix has an effect on reported results. The method requires a recovery test to be performed on a representative sample in each preparation batch, but in practice, laboratories perform recovery tests on a sample-specific basis. The HGL reviewer should verify that this QC element was evaluated in accordance with QAPP requirements.

6.3.12 Method of Standard Addition Results

The method of standard additions (MSA) is associated with GFAA analyses; this procedure is rarely performed as virtually all laboratories perform sample-specific recovery tests rather than batch-specific recovery tests. If MSA results are reported in a data package, the HGL reviewer should consult with the HGL Senior Chemist.

6.4 GENERAL CHEMISTRY

General chemistry parameters include a wide variety of analytical parameters and methodologies, including colorimetry, ion chromatography, GC, and infrared spectrometry. Usually, these parameters are secondary data that are used to determine the potential for a site to undergo monitored natural attenuation or the progress of monitored natural attenuation. Often, these tests will only require a Level II data review; however, some parameters, such as cyanide, perchlorate, anions, or total organic carbon (TOC), will on occasion require Level III validation.

In many cases, the review of general chemistry QC parameters is similar to the review of the corresponding parameters for metals. Method-specific QC parameters should be discussed in the QAPP along with the acceptance criteria and qualification requirements. Some laboratories

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SOP Category: Data Quality

Revision No.: 00
Date: November 2012

do not have summary forms for Level III QC elements and the raw data will need to be examined by the validator to evaluate performance.

The HGL reviewer should ensure that each general chemistry parameter was validated to the appropriate level, and that all appropriate QC elements were validated. If it is found that the subcontracted data validator is not applying the correct level of validation to one or more general chemistry parameters, this should be brought to the attention of the HGL Project Manager and the Project Chemist.

ATTACHMENT 3

LABORATORY QA MANUAL



Quality Systems Manual

Volume XIII, Revision I: February 2013

Effective Date: _06/10/2013
Document Control Number:
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INTRODUCTION

The Accutest Laboratories Southeast, Inc. (Accutest SE) Quality Assurance Program, detailed in this plan, has been designed to meet the quality program requirements of the National Environmental Laboratories Accreditation Conference (TNI), DoD QSM Ver 4.2, 2010 and ISO 17025. The plan establishes the framework for documenting the requirements of the quality processes regularly practiced by the Laboratory. The Quality Assurance Officer is responsible for changes to the Quality Assurance Program, which are appended to the LQSM as they occur. The plan is reviewed annually for compliance purposes by the Laboratory Director and Technical Director and edited if necessary. Changes that are incorporated into the plan are summarized in the plan introduction. Changes to the plan are communicated to the general staff in a meeting conducted by the Quality Assurance Officer following the plan's approval.

The Accutest SE plan is supported by standard operating procedures (SOPs), which provide specific operational instructions on the execution of each quality element and assure that compliance with the requirements of the plan are achieved. Accutest SE employees are responsible for knowing the requirements of the SOPs and applying them in the daily execution of their duties. These documents are updated as changes occur and the staff is trained to apply the changes.

At Accutest, we believe that satisfying client requirements and providing a product that meets or exceeds the standards of the industry is the key to a good business relationship. However, client satisfaction cannot be guaranteed unless there is a system that assures the product consistently meets its design requirements and is adequately documented to assure that all procedural steps are executed and are traceable.

This plan has been designed to assure that this goal is consistently achieved and the Accutest product withstands the rigors of scrutiny that are routinely applied to analytical data and the processes that support its generation.

Accutest Laboratories Southeast is a permanent location facility and is part of Accutest Laboratories, Inc.



Summary of Changes Accutest SE Quality System Manual –October 2012

<u>Section</u>	<u>Description</u>	Page #
Title Page	new revision number	Title
OrgChart	Lillian Torres replaced with Angel Rivera as WetChem	8
	supervisor; removed Paul Konnik from Sales.	
1	Management commitment ro constant process improvement	5
	spelled out	
16	Complete rewrite with detail and hierarchy of non-conforming products	63
App II	DoD certified methods specified in both TNI and non-TNI tables Added Perchlorate, Nitrate/Nitrite, 1,4-Dioxane,	80-83
App IV	Added 2 MS SOPs and 1 Sample Management SOP	99-101

66



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Health and Safety



1.0 QUALITY POLICY

1.1 Accutest Mission:

Accutest Laboratories provides analytical services to commercial and government clients in support of environmental monitoring and remedial activities as requested. The Laboratory's mission is dedicated to providing reliable data that satisfies clients requirements as explained in the following: "Provide easy access, high quality, analytical support to commercial and government clients which meet or exceeds data quality objectives and provides them with the data needed to satisfy regulatory requirements and/or make confident decisions on the effectiveness of remedial activities."

These services are provided impartially and are not influenced by undue commercial or financial pressures, which might impact the staff's technical judgment. Coincidentally, Accutest does not engage in activities that endanger the trust in our independent judgment and integrity in relation to the testing activities performed.

1.2 Policy Statement.

The management and staff of Accutest Laboratories share the responsibility for product quality and continually strive for its systematic improvement. Accordingly, Accutest's quality assurance program is designed to assure that all processes and procedures, which are components of environmental data production, meet established industry requirements, are adequately documented from a procedural and data traceability perspective, and are consistently executed by the staff. It also assures that analytical data of known quality, meeting the quality objectives of the analytical method in use and the data user's requirements, is consistently produced in the laboratory. This assurance enables the data user to make rational, confident, cost-effective decisions on the assessment and resolution of environmental issues.

The laboratory Quality System also provides the management staff with data quality and operational feedback information. This enables them to determine if the laboratory is achieving the established quality and operational standards, which are dictated by the client or established by regulation, such as TNI, ISO 17025 or DoD QSM. The information provided to management, through the QA program, is used to assess operational performance from a quality perspective and to perform corrective action as necessary.

All employees of Accutest Laboratories participating in environmental testing receive quality system training and are responsible for knowing and complying with the system requirements. The entire staff shares Accutest's commitment to good professional practice.

Harry Behzadi, Ph.D.

VP Southeast Operations

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Section 2: Organization



2.0 ORGANIZATION

2.1 Organizational Entity. Accutest Laboratories, Inc. is a testing laboratory founded in 1956 and registered as a New Jersey Corporation. In 2007 the laboratory has changed ownership to Accutest Holdings, Inc. Operations, staff and physical locations were not affected by the change. The laboratory headquarters are located in Dayton, New Jersey where it has conducted business since 1987. Satellite laboratories are maintained in Marlborough, Massachusetts; Orlando, Florida; San Jose, California; Denver, Colorado: Lafavette, Louisiana: and Houston, Texas.

2.2 Management Responsibilities

Requirement: Each laboratory facility will have an established chain of command. The duties and responsibilities of the management staff are linked to the President/CEO of Accutest Laboratories who establishes the agenda for all company activities.

President/CEO. Primarily responsible for all operations and business activities. Delegates authority to laboratory directors, general managers, and quality assurance director to conduct day-to-day operations and execute quality assurance duties. Each of the individual operational entities (New Jersey, Massachusetts, Florida., Texas, California, Colorado, and Louisiana) reports to the President/CEO.

Corporate Quality Assurance Director. Responsible for design, oversight, and facilitation of all quality assurance activities established by the Quality Program. Directly reports to the President/CEO.

Vice President Operations/Laboratory Director. There is a Laboratory Director assigned to each of the following operational entities: New Jersey, Massachusetts Florida, Louisiana, and West (Texas, California, and Colorado). The Laboratory Director executes day-to-day responsibility for laboratory operations including technical aspects of production activities and associated logistical procedures. Directly reports to the President/CEO.

Quality Assurance Officer (on location). Responsible for oversight, implementation and facilitation of all quality assurance activities established by the Quality Program. Directly reports to the Laboratory Director. Also exchanges information with and submits laboratory performance data (PE scores, audit reports, accreditation changes, etc.) to Corporate QA Director. Takes program directions from Corporate QA Director.

Technical Director. Responsible for oversight and implementation of technical aspects of production activities in the environmental testing laboratory. In the event that the technical director, quality assurance director, or laboratory manager is absent for a period of time that exceeds 15 consecutive calendar days, the designated appointees shall temporarily perform the technical director, quality assurance director, or laboratory manager's job function. If this absence exceeds 65 consecutive calendar days, the Accreditation Body(ies), including DoD ELAP, is to be notified in writing.



Current list of appointed deputies located in restricted access controlled document directory

Department Managers. Executes day-to-day responsibility for specific laboratory areas including technical aspects of production activities and associated logistical procedures. Directly report to the Laboratory Director.

Section Supervisors. Executes day-to-day responsibility for specific laboratory units including technical aspects of production activities and associated logistical procedures. Directly report to the Department Manager.

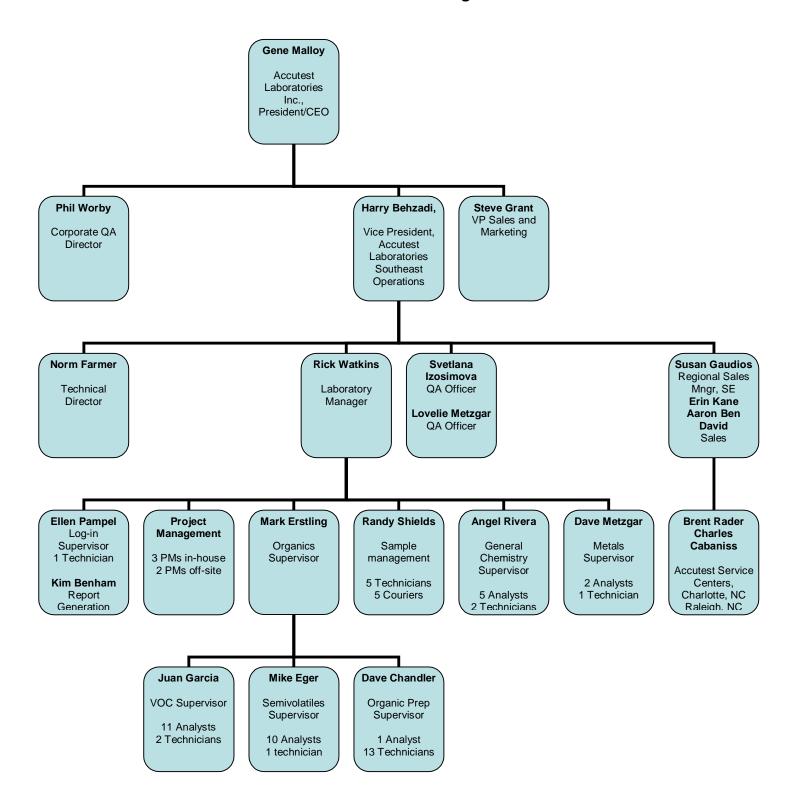
2.3 Chain of Command

The responsibility for managing all aspects of the Company's operation is delegated to specific individuals, who have been assigned the authority to act in the absence of the senior staff. These individuals are identified in the following Chain of Command:

Harry Behzadi, Ph.D., VP, Southeast Operations Norm Farmer, Technical Director (Operations and IT) Rick Watkins, Laboratory Manager (Operations) Heather Wandrey, Project Manager (Client Services)



Accutest Laboratories Southeast Organizational Chart





3.0 QUALITY RESPONSIBILITIES OF THE MANAGEMENT TEAM

Requirement: Each member of the management team has a defined responsibility for the Quality Program. Program implementation and operation is designated as an operational management responsibility. Program design and implementation is designated as a Quality Assurance Responsibility.

President/CEO: Primary responsibility for all quality activities. Delegates program responsibility to the Quality Assurance Director. Serves as the primary alternate in the absence of the Quality Assurance Director. Has the ultimate responsibility for implementation of the Quality Program.

Vice President Operations/Laboratory Director. Responsible for implementing and operating the Quality Program in all laboratory areas. Responsible for the design and implementation of corrective action for defective processes. Has the authority to delegate Quality Program implementation responsibilities.

Corporate Quality Assurance Director. Responsible for design, implementation support, training, and monitoring of the quality system. Identifies product, process, or operational defects using statistical monitoring tools and processes audits for elimination via corrective action. Empowered with the authority to halt production if warranted by quality problems. Monitors implemented corrective actions for compliance.

Quality Assurance Officer (on location). Responsible for design support, implementation support, and monitoring support of the quality system. Training personnel in various aspects of quality system. Conducts audits and product reviews to identify product, process, or operational defects using statistical monitoring tools and processes audits for elimination via corrective action. Empowered with the authority to halt production if warranted by quality problems. Monitors implemented corrective actions for compliance.

Technical Director. Responsible for oversight and implementation of technical aspects of Quality System as they are integrated into method applications and employed to assess analytical controls on daily basis. The Technical Director reviews and acknowledges the technical feasibility of proposed quality system involving technical applications.

Department Managers. Responsible for applying the requirements of the Quality Program in their section and assuring subordinate supervisors and staff apply all program requirements. Initiates, designs, documents, and implements corrective action for quality deficiencies.

Group Leaders. Responsible for applying the requirements of the Quality Program to their operation and assuring the staff applies all program requirements. Initiates, designs, documents, and implements corrective action for quality deficiencies.



Bench Analysts. Responsible for applying the requirements of the Quality Program to the analyses they perform, evaluating QC data and initiating corrective action for quality control deficiencies within their control. Implements global corrective action as directed by superiors.

3.2 **Program Authority**:

Authority for program implementation on corporate level originates with the President/CEO who bears ultimate responsibility for program design, implementation, and enforcement of requirements. This authority and responsibility is delegated to the Director of Quality Assurance who performs quality functions independently without the encumbrances or biases created by operational or production responsibilities to ensure an honest, independent assessment of quality issues.

Laboratory Director and Quality Assurance Officer mirror this authority on location.

3.3 **Data Integrity Policy**:

The Accutest Data Integrity Policy reflects a comprehensive, systematic approach for assuring that data produced by the laboratory accurately reflects the outcome of the tests performed on field samples and has been produced in a bias free environment by ethical professionals. The policy includes a commitment to technical ethics, staff training in ethics and data integrity, an individual attestation to data integrity and procedures for evaluating data integrity. Senior management assumes the responsibility for assuring compliance with all technical ethics elements and operation of all data integrity procedures. The staff is responsible for compliance with the ethical code of conduct and for practicing data integrity procedures.

The Accutest Data Integrity Policy is as follows:

"Accutest Laboratories is committed to producing data that meets the data integrity requirements of the environmental regulatory community. This commitment is demonstrated through the application of a comprehensive data integrity program that includes ethics and data integrity training, data integrity evaluation procedures, staff participation and management oversight. Adherence to the specifications of the program assures that data provided to our clients is of the highest possible integrity and can be used for decision making processes with high confidence."



Data Integrity Responsibilities

Management. Senior management retains oversight responsibility for the data integrity program and retains ultimate responsibility for execution of the data integrity program elements. Senior management is responsible for providing the resources required to conduct ethics training and operate data integrity evaluation procedures. They also include responsibility for creating an environment of trust among the staff and being the lead advocate for promoting the data integrity policy and the importance of technical ethics.

Staff. The staff is responsible for adhering to the company ethics policy as they perform their duties and responsibilities associated with sample analysis and reporting. By executing this responsibility, data produced by Accutest Laboratories retains its high integrity characteristics and withstands the rigors of all data integrity checks.

The staff is also responsible for adhering to all laboratory requirements pertaining to manual data edits, data transcription and data traceability. These include the application of approved manual peak integration and documentation procedures. It also includes establishing traceability for all manual results calculations and data edits.

Ethics Statement. The Accutest ethics statement reflects the standards that are expected for businesses that provide environmental services to regulated entities and regulatory agencies on a commercial basis. The Ethics Policy is comprised of key elements that are essential to organizations that perform chemical analysis for a fee. As such, it focuses on elements related to personal, technical and business activities.

Accutest Laboratories provides analytical chemistry services on environmental matters to the regulated community. The data the company produces provides the foundation for determining the risk presented by a chemical pollutant to human health and the environment. The environmental industry is dependent upon the accurate portrayal of environmental chemistry data. This process is reliant upon a high level of scientific and personal ethics.

It is essential to the Company that each employee understands the ethical and quality standards required to work in this industry. Accordingly, Accutest has adopted a code of ethics, which each employee is expected to adhere to as follows:

- Perform chemical and microbiological analysis using accepted scientific practices and principles.
- Perform tasks in an honest, principled and incorruptible manner inspiring peers & subordinates.
- Maintain professional integrity as an individual.
- Provide services in a confidential, honest, and forthright manner.
- Produce results that are accurate and defensible.



- Report data without any considerations of self-interest.
- Comply with all pertinent laws and regulations associated with assigned tasks and responsibilities.

Data Integrity Procedures.

Four key elements comprise the Accutest data integrity system:

- 1) data integrity training,
- 2) signed data integrity documentation for all laboratory employees,
- 3) in-depth, periodic monitoring of data integrity, and
- 4) data integrity procedure documentation.

Procedures have been implemented for conducting data integrity training and for documenting that employees conform to the Accutest Data Integrity and Ethics policy.

The data integrity program consists of routine data integrity evaluation and documentation procedures to periodically monitor and document data integrity. These procedures are documented in SOPs. SOPs are approved and reviewed annually following the procedures employed for all Accutest SOPs. Documentation associated with data integrity evaluations is maintained on file and is available for review.

Data Integrity Training, .Accutest employees receive technical ethics training during new employee orientation. Employees are also required to attend annual ethics refreshment training and sign an ethical conduct agreement annually, which verifies their understanding of Accutest's technical ethics policy and their ethical responsibilities. The agreement is refreshed annually and appended to each individual's training file.

The training focuses on the reasons for technical ethic training, explains the impact of data fraud on human health and the environment, and illustrates the consequences of criminal fraud on businesses and individual careers. Multiple examples of prohibited practices are reviewed and discussed. Accutest's ethics policy and code of ethics are reviewed and explained for each new employee. Employees receive Accutest's technical ethics brochure for further review.

Training on department-specific data integrity procedures are conducted by individual departments for groups involved in data operations. These include procedures for manual chromatographic peak integration, standards traceability, etc.

Data Integrity Training Documentation. Records of all data integrity training are maintained in individual training folders. Attendance at all training sessions is documented and appended to the training file.

Accutest Data Integrity and Ethical Conduct Agreement. All employees are required to sign a Data Integrity and Ethical Conduct Agreement annually. This document is archived in individual training files, which are retained for duration of employment.



The Data Integrity and Ethical Conduct Agreement is as follows:

- I. I understand the high ethical standards required of me with regard to the duties I perform and the data I report in connection with my employment at Accutest Laboratories.
- II. I have received formal instruction on the code of ethics that has been adapted by Accutest Laboratories and agree to comply with these requirements.
- III. I have received formal instruction on the elements of Accutest Laboratories' Data Integrity Policy and have been informed of the following specific procedures:
 - a. Routine data integrity monitoring is conducted on sample data, which may include an evaluation of the data I produce,
 - b. Formal procedures for the confidential reporting of data integrity issues are available, which can be used by any employee,
 - c. A data integrity investigation is conducted when data issues are identified that may negatively impact data integrity.
- IV. I am aware that data fraud is a punishable crime that may include fines and/or imprisonment upon conviction.
- V. I also agree to the following:
 - a. I shall not intentionally report data values, which are not the actual values observed or measured.
 - b. I shall not intentionally modify data values unless the modification can be technically justified through a measurable analytical process.
 - c. I shall not intentionally report dates and times of data analysis that are not the true and actual times the data analysis was conducted.
 - d. I shall not condone any accidental or intentional reporting of inauthentic data by other employees and immediately report it's occurrence to my superiors.
 - e. I shall immediately report any accidental reporting of inauthentic data by myself to my superiors.

Data Integrity Monitoring. Several documented procedures are employed for performing data integrity monitoring. These include regular data review procedures by supervisory and management staff (Section 12.7), supervisory review and approval of manual integrations and periodic reviews of data audit trails from the LIMS and all computer controlled analysis.



Data Review. All data produced by the laboratory undergoes several levels of review, which includes two levels of management review. Detected data anomalies that appear to be related to data integrity issues are isolated for further investigation. The investigation is conducted following the procedures described in this section.

Manual Peak Integration Review and Approval. Routine data review procedures for all chromatographic processes includes a review of all manual chromatographic peak integrations. This review is performed by the management staff and consists of a review of the machine integration compared to the manual integration. Manual integrations, which have been performed in accordance with Accutest's manual peak integration procedures are approved for further processing and release. Manual integrations which are not performed to Accutest's specifications are set aside for corrective action, which may include analyst retraining or further investigation as necessary.

Data Audit Trail Review. Data integrity audits are comprehensive data package audits that include a review of raw data, process logbooks, processed data reports and data audit trails from individual instruments and LIMS. Data audit trails, which record all electronic data activities, are available for the majority of computerized methodology and the laboratory information management system (LIMS). These audit trails are periodically reviewed to determine if interventions performed by technical staff constitute an appropriate action. The review is performed on a recently completed job and includes interviews with the staff that performed the analysis. Findings indicative of inappropriate interventions or data integrity issues are investigated to determine the cause and the extent of the anomaly.

Confidential Reporting Of Data Integrity Issues. Data integrity concerns may be raised by any individual to their supervisor. Employees with data integrity concerns should always discuss those concerns with their immediate supervisors as a first step unless the employee is concerned with the confidentiality of disclosing data integrity issues or is uncomfortable discussing the issue with their immediate supervisors. The supervisor makes an initial assessment of the situation to determine if the concern is related to a data integrity violation. Those issues that appear to be violations are documented by the supervisor and referred to the QA Officer (local) for investigation.

Documented procedures for the confidential reporting of data integrity issues in the laboratory are part of the data integrity policy. These procedures assure that laboratory staff can privately discuss ethical issues or report items of ethical concern without fears of repercussions with senior staff.

Employees with data integrity concerns that they consider to be confidential are directed to the Corporate Human Resources Manager in Dayton, New Jersey. The HR Manager acts as a conduit to arrange a private discussion between the employee and the Corporate QA Director or a local QA Officer.

During the employee - QA discussion, the QA representative evaluates the situation presented by the employee to determine if the issue is a data integrity concern or a legitimate practice. If the practice is legitimate, the QA representative clarifies the



process for the employee to assure understanding. If the situation appears to be a data integrity concern, the QA representative initiates a Data Integrity Investigation following the procedures specified in SOPs QA038-QA041.

Data Integrity Investigations. Follow-up investigations are conducted for all reported instances of ethical concern related to data integrity. Investigations are performed in a confidential manner by senior management according to a documented procedure. The outcome of the investigation is documented and reported to the company president who has the ultimate responsibility for determining the final course of action in the matter. Investigation documentation includes corrective action records, client notification information and disciplinary action outcomes, which is archived for a period of five years.

The investigations are conducted by the senior staff and supervisory personnel from the affected area. The investigation team includes the Laboratory Director and the Quality Assurance Officer. Investigations are conducted in a confidential manner until it is completed and resolved.

The investigation includes a review of the primary information in question by the investigations team. The team performs a review of associated data and similar historical data to determine if patterns exist. Interviews are conducted with key staff to determine the reasons for the observed practices.

Following data compilation, the investigations team reviews all information to formulate a consensus conclusion. The investigation results are documented along with the recommended course of action.

Corrective Action, Client Notification & Discipline. Investigations that reveal systematic data integrity issues will go through corrective action for resolution and disposition (Section 13). If the investigation indicates that an impact to data has occurred and the defective data has been released to clients, client notification procedures will be initiated following the steps in Section 17.6.

In all cases of data integrity violations, some level of disciplinary action will be conducted on the responsible individual. The level of discipline will be consistent with the violation and may range from retraining and/or verbal reprimand to termination.



4.0 JOB DESCRIPTIONS OF KEY STAFF

Requirement: Descriptions of key positions within the organization must be defined to ensure that clients and staff understand duties and the responsibilities of the management staff and the reporting relationships between positions.

President/Chief Executive Officer. Responsible for all laboratory operations and business activities. Establishes the company mission and objectives in response to business needs. Direct supervision of the Vice President of Operations, each laboratory director, client services, management information systems, and quality assurance.

Vice President, Operations/Laboratory Director. Reports to the company president. Establishes regional laboratory operations strategy and business development. Authorized to enter into contractual agreements on Company's behalf.

Director, Quality Assurance. Reports to the company president. Establishes the company quality agenda, develops quality procedures, provides assistance to operations on quality procedure implementation, coordinates all quality control activities monitors the quality system and provides quality system feedback to management to be used for process improvement.

Vice President, Information Technlogies Reports to the company president. Develops the MIS software and hardware agenda. Provides system strategies to compliment company objectives. Maintains all software and hardware used for data handling.

Client Services, Sales, Account Manager(s). Reports to the company president. Establishes and maintains communications between clients and the laboratory pertaining to client requirements which are related to sample analysis and data deliverables. Initiates client orders and supervises sample login operations.

Quality Assurance Officer (on location). Reports to the Laboratory Director. Develops quality procedures, provides assistance to operations on quality procedure implementation, coordinates all quality control activities, monitors the quality system, and provides quality system feedback to management to be used for process improvement. In the event of prolonged absence QAO also designated a Deputy Technical Director, unless otherwise specified by internal memo from Laboratory Director.

Manager Client Services (on location). Reports to the Laboratory Director. Establishes and maintains communications between clients and the laboratory pertaining to client requirements which are related to sample analysis and data deliverables. Initiates client orders and supervises sample login operations.

Technical Director (On Location). Reports to the laboratory director. Establishes laboratory operations strategy. Direct supervision of organic chemistry and inorganic



chemistry. Directs the operations, preparation and instrumental analysis. Responsible for following Quality Program requirements. Assumes operational responsibilities of Lab Director in his absence.

Laboratory Manager. Reports to the Laboratory Director. Directs the day-to day operations of entire laboratory, direct supervision of organic chemistry, inorganic chemistry, field services, and sample management.

Oversees daily work schedule as developed by respective departments. Supervises method implementation. Responsible for following Quality Program requirements. Maintains laboratory instrumentation in an operable condition.

Supervisors, Shipping and Receiving Departments. Reports to the Laboratory Manager. Develops, maintains and executes all procedures required for transport and receipt of samples, verification of preservation, and chain of custody documentation. Responsible for maintaining and documenting secure storage, delivery of samples to laboratory units on request, and disposal following completion of all analytical procedures.

Supervisor, Wet Chemistry. Reports to the Laboratory Manager. Directs the operations of the wet chemistry group. Establishes and executes daily work schedule. Supervises method implementation, application, and data production. Supervises the analysis of samples for wet chemistry parameters using valid, documented methodology. Maintains instrumentation in an operable condition. Reviews data for compliance to quality and methodological requirements. Responsible for following Quality Program requirements.

Supervisor, Metals. Reports to the Laboratory Manager. Directs the operations of the metals group. Establishes and executes daily work schedule. Supervises method implementation, application, and data production. Supervises the analysis of samples for metallic elements using valid, documented methodology. Documents all procedures and data production activities. Maintains instrumentation in an operable condition. Reviews data for compliance to quality and methodological requirements. Responsible for following Quality Program requirements

Supervisor, Organic Preparation. Reports to the Laboratory Manager. Directs the operations of the sample preparation group. Establishes and executes daily work schedule. Supervises method implementation, and application. Supervises the preparation of samples for organic compounds using valid, documented methodology. Documents all procedures and data production activities. Maintains laboratory equipment in an operable condition. Reviews records for compliance to quality and methodological requirements. Responsible for following Quality Program requirements.

Volatile and Semivolatie Supervisors, Organics. Reports to the Laboratory Manager. Directs the operations of the respective organics group. Establishes and executes daily work schedule. Supervises method implementation, application, and data production. Supervises the analysis of samples for organic compounds using valid, documented methodology. Documents all procedures and data production



activities. Maintains instrumentation in an operable condition. Reviews data for compliance to quality and methodological requirements. Responsible for following Quality Program requirements

Report Generation Supervisor. Reports to Laboratory Manager. Oversees report generation and fulfillment of client specifications as applied to data deliverables. Responsible for data delivery in timely manner.

Detailed Job descriptions of lab personnel are found in training folders

4.2 Employee Screening, Orientation, and Training.

All potential laboratory employees are screened and interviewed by human resources and technical staff prior to their hire. The pre-screen process includes a review of their qualifications including education, training and work experience to verify that they have adequate skills to perform the tasks of the job. Minimum qualifications for non-technical personnel require High School diploma (couriers also shall posses clean driving record), technical personnel must also demonstrate basic laboratory experience, such as balance and syringe use, aseptic practices, etc. College-level science coursework is favored.

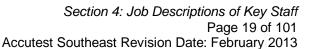
Newly hired employees receive orientation training beginning the first day of employment by the Company. Orientation training consists of initial health and safety training and a detailed review of the personal protection policies, technical ethics training and data integrity procedures and quality assurance program training (including Company's goals, objectives, mission, and vision).

All technical staff receives training to develop and demonstrate proficiency for the methods they perform. New analysts work under supervision until the supervisory staff is satisfied that a thorough understanding of the method is apparent. Organics/Inorganics analysts are required to demonstrate method proficiency through a precision and accuracy study. Data from the study is compared to method acceptance limits. If the data is unacceptable, additional training is required. The analyst must also demonstrate the ability to produce acceptable data through the analysis of an independently prepared proficiency sample.

Proficiency is demonstrated annually. Data from initial and continuing proficiency demonstration is archived in the individual's training folder. In the instance where analyte can not be spiked in the clean matrix, such as TSS or pH, the results of an external Performance Evaluation (PE) sample may be used to document analyst's proficiency.

Minimum training required for administrative staff consists of laboratory safety and ethical conduct.

Training Documentation. The QA Officer prepares a training file for every new employee. All information related to qualifications, experience, external training courses, and education are placed into the file. Verification documentation for





orientation, health & safety, quality assurance, and ethics training is also included in the file.

Additional training documentation is added to the file as it occurs. This includes data for initial and continuing demonstrations of proficiency, performance evaluation study data and notes and attendance lists from group training sessions.

The Quality Assurance Department maintains the employee training database. This database is a comprehensive inventory of training documentation for each individual employee. The database enables supervisors to obtain current status information on training data for individual employees on a job specific basis. It also enables the management staff to identify training documentation in need of completion.

Employee specific database records are created by human resources on the date of hire. Data base fields for job specific requirements such as SOP documentation of understanding and annual demonstration of analytical capability are automatically generated when the supervisor assigns a job responsibility. Employees acknowledge that their SOP responsibilities have been satisfied using a secure electronic process, which updates the database record. Reports are produced which summarize the qualifications of individual employees or departments.



Accutest Southeast Revision Date: February 2013

5.0 SIGNATORY APPROVALS

Requirement: Procedures are required for establishing the traceability of data and documents. The procedure consists of a signature hierarchy, indicating levels of authorization for signature approvals of data and information within the organization. Signature authority is granted for approval of specific actions based on positional hierarchy within the organization and knowledge of the operation that requires signature approval. A log of signatures and initials of all employees is maintained for cross-referencing purposes.

5.1 Signature Hierarchy.

President/Chief Executive Officer. Authorization for contracts and binding agreements with outside parties. Approval of final reports, quality assurance policy, SOPs, project specific QAPs, data review and approval in lieu of technical managers. Contract signature authority resides with Company Officers only, which include the President/CEO, CFO and VP Administration.

Vice President, Operations/Laboratory Director. Approval of final reports and quality assurance policy in the absence of the President. Approval of SOPs, project specific QAPs, data review and approval in lieu of technical managers. Technical policy.

Technical Director (on location): Approval of final reports and quality assurance policy in the absence of the Laboratory Director. Approval of SOPs, project specific QAPs, data review and approval in lieu of technical managers. Technical policy review. In the event of prolonged absence refer to list of approved deputies – sec 2.2.

Director, Quality Assurance. Approval of final reports and quality assurance policy in the absence of the President. Approval of SOPs, project specific QAPs, data review and approval in lieu of technical managers.

Quality Assurance Officer (on location). Approval of final reports and quality assurance policy in the absence of the Laboratory Director. Approval of SOPs, project specific QAPs, data review and approval in lieu of technical managers. In the event of prolonged absence refer to list or appointed deputies – see sec. 2.2.

Manager, Sample Management. Initiation of laboratory sample custody and acceptance of all samples. Approval of department policies and procedures. Department specific supplies purchase. Waste manifesting and disposal.

Project Manager, Client Services. QAP and sampling and analysis plan approval. Project specific contracts, pricing, and price modification agreements. Approval and acceptance of incoming work, Client services policy.







Supervisors, Technical Departments. Methodology and department specific QAPs. Data review and approval, department specific supplies purchase. Technical approval of SOPs.

Supervisors, Technical Departments. Data review approval, purchasing of expendable supplies.

- 5.2 <u>Signature Requirements</u>. All laboratory activities related to sample custody and generation or release of data must be approved using either initials or signatures. The individual, who applies his signature or initial to an activity or document, is authorized to do so within the limits assigned to them by their supervisor. All signatures and initials must be applied in a readable format that can be cross-referenced to the signatures and initials log if necessary.
- 5.3 <u>Signature and Initials Log</u>. The QA Officer maintains a signature and initials log. New Employee signatures and initials are appended to the log on the first day of employment. Signature of individuals no longer employed by the company are retained.





6.0 DOCUMENTATION and DOCUMENT CONTROL

Requirement: Document control policies have been established which specify that any document used as an information source or for recording analytical or quality control information must be managed using defined document control procedures. Accordingly, policies and procedures required for the control, protection, and storage of any information related to the production of analytical data and the operation of the quality system to assure its integrity and traceability have been established and implemented in the laboratory. The system contains sufficient controls for managing, archiving and reconstructing all process steps, which contributed to the generation of an analytical test result. Using this system, an audit trail for reported data can be produced, establishing complete traceability for the result.

6.1 Administrative Records. The Quality Assurance Officer manages Administrative (non-analytical) records. These records consist of electronic documents that are retained in a limited access electronic directory, which are released to the technical staff upon specific request.

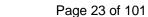
<u>Form Generation & Control</u>. The Quality Assurance Officer approves all forms used as either stand-alone documents or in logbooks to ensure their traceability. Forms are generated as computer files only and maintained in a limited access master directory. Access to the electronic forms and applications is granted to QA Officer, Laboratory Manager and Technical Director(s) (local and regional). Approved forms must display the date of current revision and initials of person who revised the form. Modifications to existing forms are approved by QA, obsolete forms moved to archive directory and retained for minimum of five years.

New forms must include Accutest SE identification and appropriate spaces for signatures of approvals and dates. Further design specifications are the responsibility of the originating department.

Technical staff is required to complete all forms to the maximum extent possible. If information for a specific item is unavailable, the analyst is required to cross out the information block. The staff is also required to cross out the uncompleted portions of a logbook or logbook form if the day's analysis does not fill the entire page of the form.

<u>Logbook Control</u>. All laboratory logbooks are controlled documents that are comprised of approved forms used to document specific processes. Logbook control is maintained by QA staff.

New logs are numbered and issued to a specific individual who is assigned responsibility for the log. Supervisor performs periodical review of the logbooks. Old logs are returned to QA for entry into the document archive system where they are retained for minimum of five (5) years. Laboratory staff may hold a maximum of two consecutively dated logbooks of the same type in the laboratory, not including the most recently issued book to simplify review of recently completed analysis.





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Controlled Documents. Key laboratory documents are designated for controlled document status to assure that identities of individuals receiving copies and the number of copies that have been distributed are known. Controlled status simplifies document updates and retrieval of outdated documents. Control is maintained through a document numbering procedure and document control logbook designating the individual receiving the controlled document. Document control is also maintained by pre-designating the numbers of official copies of documents that are placed into circulation within the laboratory.

Quality Systems Manual (QSM). All QSMs are assigned a number prior to distribution. The QSMs are distributed as controlled documents i.e. ones that will be collected back and replaced with next version (documents distributed to the Accutest Inc. staff). QSMs distributed to outside entities are considered tracked documents since there is no possibility of collecting them back and ensuring that current revision is in use. These situation include bid submissions, client requests, etc. These copies are watermarked as "Uncontrolled Documents" The control/tracking number, date of distribution, and identity of the individual receiving the document are recorded in the document control spreadsheet. QA staff maintains tracking spreadsheet. The numbering system is continuous.

Standard Operating Procedures (SOPs). SOPs are maintained by pre-designating the numbers of official copies of documents that are placed into circulation within the laboratory. Official documents are printed and placed into the appropriate laboratory section as follows:

Sample Management: One copy for the sample receiving file Bottle preparation area – One copy for shipping area Organics Laboratories: One for the affected laboratory area.

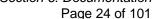
Inorganics Laboratories: One for the affected laboratory area.

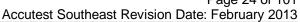
The original, signed copy of the SOP is maintained in the master SOP binder by the QA staff.

Documents are controlled using an "Official Copy" stamp in red ink. Additional copies could be issued to individuals for training purposes. Distribution is documented on SOP cover page. Superceded copies collection is conducted accordingly to cover page distribution list.

SOPs distributed to clients as part of bid submission, pre-audit evaluation, etc. are watermarked as "Proprietary Information".

Quick reference cards: These documents are compiled for lab staff convenience and are based on current SOP revision and/or recent regulatory updates. These one- or two-sided documents are footnoted with reference to SOP/regulatory standard. stamped with "Official Copy" stamp in red ink and laminated for durability. Use of these quick references does not substitute reading and acknowledging the parent SOP.







Operators' Manuals are considered controlled documents and stored in appropriate departments.

6.2 **Technical Records.** All records related to the analysis of samples and the production of analytical results are archived in secure document storage or on electronic media and contain sufficient detail to produce an audit trail, which re-creates the analytical result. These records include information related to the original client request, bottle order, sample login and custody, storage, sample preparation, analysis, data review and data reporting.

Records that can not be maintained on electronic media are considered irretrievable records, segregated into separate secured storage and access controlled with access log maintained by QA Staff. Examples of such records are employee training files, obsolete SOPs and acknowledgement form originals, training files, logbooks, etc.

Each department involved in this process maintains controlled documents, which enable them to maintain records of critical information relevant to their department's process.

6.3 Quality Assurance Directory. All Quality Assurance documentation and quality control limit data is stored in a restricted QA directory on the network server. The directory has been designated as read only. The QA staff, technical director and the laboratory manager have write capability in this directory. Information on this directory is backed-up daily.

This directory contains all current and archived Quality System Manuals, SOPs, control limits, MDL studies, precision and accuracy data, internal and external audit reports, official forms, Health and Safety materials, PT scores, State Certifications and metrics calibration information.

6.4 Analytical Records. All data related to the analysis of field samples are retained as either paper or electronic records that can be retrieved to compile a traceable audit trail for any reported result. All information is linked to the client job and sample number, which serves as a reference for all sample related information tracking.

Critical times in the life of the sample from collection through analysis to disposal are documented. This includes date and time of collection, receipt by the laboratory, preparation times and dates, analysis times and dates and data reporting information. Analysis times are calculated in hours for methods where holding time is specified in hours (≤72 hours).

Sample preparation information is recorded in a separate controlled logbook or on controlled forms in three-ring binder. It includes sample identification numbers, types of analysis, preparation and cleanup methods, sample weights and volumes, reagent lot numbers and volumes and any other information pertinent to the preparation procedure.





Information related to the identification of the instrument used for analysis is permanently attached to the electronic record. The record includes an electronic data file that indicates all instrument conditions employed for the analysis, including the type of analysis conducted. The analyst's identification is electronically attached to the record. The instrument tuning and calibration data is electronically linked to the sample or linked though paper logs, which were used in the documentation of the analysis. Quality control and performance criteria are permanently linked to the paper archive or electronic file.

Paper records for the identity, receipt, preparation and evaluation of all standards and reagents used in the analysis are documented in prepared records and maintained in controlled documents or files. Lot number information linking these materials to the analysis performed is recorded in the logbooks associated with the samples in which they were used.

Manual calculations or peak integrations that were performed during the data review are retained as paper or electronically generated PDF documents and included as part of the electronic archive. Signatures for data review are retained on paper or as electronic stamps on PDF versions of the paper record for the permanent electronic file.

Confidential Business Information (CBI). Operational documents including SOPs, Quality Manuals, personnel information, internal operations statistics, and laboratory audit reports are considered confidential business information. Strict controls are placed on the release of this information to outside parties.

Release of CBI to outside parties or organizations may be authorized upon execution of a confidentiality agreement between Accutest and the receiving organization or individual. CBI information release is authorized for third party auditors and commercial clients in electronic mode as Adobe Acrobat .PDF format only.

- 6.6 <u>Software Change Documentation & Control.</u> Changes to software are documented as text within the code of the program undergoing change. Documentation includes a description of the change, reason for change and the date the change was placed into effect. Documentation indicating the adequacy of the change is prepared following the evaluation by the user who requested the change.
- 6.7 Report and Data Archiving. Accutest Laboratories maintains electronic image file copies of original reports in archive for a minimum period of five (5) years. After five years, the files are automatically discarded unless contractual arrangements exist which dictate different requirements. Client specific data retention practices are employed for government organizations such as the Department of Defense Agencies and MA DEP that require a retention period of ten (10) years, as well as commercial clients upon contractual requirements agreement.

Complete date and time stamped client reports are generated from LIMS using the source documents archived on Document server. These source documents are maintained on document server and backed up to primary and clone tapes. Accutest





archives the original report (organized by job number) and the organic and inorganic support data. Organic support data is archived according to instrument batch numbers. All organics data is backed up to the tape or archive drive via Networker Backup software and/or AccuBack backup software. Data from the archive drive is then written to tape at periodic intervals.

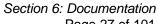
Wet chemistry support data is archived by analytical batch (GN...). Metals support data is archived by instrument batch (MA...). Metals digestion data is archived as digestion logbooks.

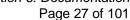
The reports generation group electronically scans completed reports and stores them by job number on the document server. The document server is backed up daily to a digital tape. Copies of these files remain active on the document server for easy review access. The digital tapes remain in secure storage for the remainder of the archive period.

6.8 <u>Training</u>. Ongoing training ensures competence of all relevant personnel. At the minimum personnel should possess knowledge of the technology used in the testing, general requirements expressed in legislature and industry standards, and understand the significance of deviations with regard to approved procedures. The company maintains a training record for all employees that documents that they have received instruction on administrative and technical tasks that are required for the job they perform. Training records for individuals employed by the company are retained for a period of five years following their termination of employment.

<u>Training File Origination</u>. The Quality Assurance Officer (QAO) initiates training files. Quality Assurance officer retains the responsibility for the maintenance and tracking of all training related documentation in the file. The file is begun on the first day of employment. Information required for the file includes a copy of the individual's most current resume, detailing work experience and a copy of any college diplomas or transcript(s). Information added on the first day includes documentation of health and safety training and a signed Ethics and Data Integrity agreement. These two constitute minimal necessary training for Project Management and Administrative staff. Training documentation, training requirements, analyst proficiency information and other training related support documentation is tracked using a customized database application. Database extracts provide an itemized listing of specific training requirements by job function. Training status summaries for individual analysts portray dates of completion for job specific training requirements.

Technical Training. The supervisor of each new employee is responsible for developing a training plan for each new employee. The supervisor updates the outline, adding signatures and dates as training elements are completed at regular frequency. Supporting documentation, such as precision and accuracy studies, which demonstrate analyst capability for a specific test, are added as completed. When analyte can not be spiked, such as pH or TSS, external PE sample is purchased and analyzed. Where no external PE sample is available, sample duplicates must be successfully analyzed. Method review records are retained where analysis of duplicates is not possible. Employees and supervisors verify documentation of

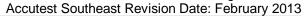








understanding (DOU) for all assigned standard operating procedures in the training database. Certificates or diplomas for any off-site training are added to the file.





7.0 REFERENCE STANDARD TRACEABILITY

<u>Requirement</u>: Documented procedures, which establish traceability between any measured value and a national reference standard, must be in place in the laboratory. All metric measurements must be traceable to NIST reference weights or thermometers that are calibrated on a regular schedule. All chemicals used for calibration of a quantitative process must be traceable to an NIST reference that is documented by the vendor using a certificate of traceability. The laboratory maintains a documentation system that establishes the traceability links. The procedures for verifying and documenting traceability must be documented in standard operating procedures.

- 7.1 Traceability of Metric Measurements - Thermometers. Accutest uses NISTtraceable thermometers to calibrate commercially purchased working laboratory thermometers prior to their use in the laboratory and annually thereafter for liquid in glass thermometers or quarterly for electronic temperature measuring devices. If necessary, these working thermometers are assigned correction factors that are determined during their calibration using an NIST-traceable thermometer as the standard. The correction factor is documented in a thermometer log and on a tag attached to the working thermometer. Both original observation and corrected measurement are recorded in the temperature log. The NIST-traceable reference thermometer is checked for accuracy by an outside vendor minimum every five (5) years following the specifications for NIST-traceable thermometer calibration verification detailed in the United States Environmental Protection Agency's "Manual for the Certification of Laboratories Analyzing Drinking Water", Fifth Edition, January 2005. Currently the NIST thermometer is verified by outside vendor on triennial basis due to contract-specific requirements. Calibration log and Certificate(s) of calibration are maintained on file with QAO.
- 7.2 <u>Traceability of Metric Measurements Calibration Weights</u>. Accutest uses calibrated weights, which are traceable to NIST standard weights to calibrate all balances used in the laboratory. Balances must be calibrated to specific tolerances within the intended use range of the balance. Calibration checks are required on each day of use. If the tolerance criteria are not achieved, corrective action specified in the balance calibration SOP must be applied before the balance can be used for laboratory measurements. All weights are recalibrated by outside vendor every five years following the specifications for weight calibration verification detailed in the United States Environmental Protection Agency's "Manual for the Certification of Laboratories Analyzing Drinking Water", Fifth Edition, January 2005. Certificate(s) of calibration are maintained on file with QAO. Balances are inspected and maintained by professional service technicians annually. Certificate(s) of inspection are maintained with QAO.
- 7.3 <u>Traceability of Chemical Standards and Reagents</u>. All chemicals and reagents, with the exception of bulk dry Na2SO4 and solvents purchased as reference standards for use in method calibration must establish traceability to NIST referenced material through a traceability certificate (Certificate of Analysis, CoA). Process links are





established that enable a calibration standard solution to be traced to its NIST reference certificate. Solvents, acids and other supplies are being tested to verify their suitability for the analytical process.

7.4 Assignment Of Reagent and Standard Expiration Dates. Expiration date information for all purchased standards and reagents is provided to Accutest with all prepared standard solutions and unstable reagents as a condition of purchase. Neat materials and inorganic reagents are not required to be purchased with expiration dates. Certified prepared solutions are labeled with the expiration date provided by the manufacturer. In-house prepared solutions are assigned expiration dates that are consistent with the method that employs their use unless documented experience indicates that an alternate date can be applied. If alternate expiration dates are employed, their use is documented in the method SOP. Expiration dates for prepared inorganic reagents, which have not exhibited instability, are established at two years form the date of preparation for tracking purposes. All containers shall be labeled with the date of preparation and expiration date clearly indicated.

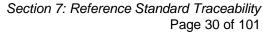
The earliest expiration date is always the limiting date for assigning expiration dates to prepared solutions. Expiration dates that are later than the expiration date of any derivative solution or material are prohibited.

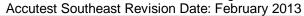
7.5 <u>Documentation of Traceability</u>. Traceability information is documented in individual logbooks designated for the measurement process in use. The QA Officer maintains calibration documentation for metric references in pertinent folders and logbooks.

Balance calibration verification is documented in logbooks that are assigned to each balance. The individual conducting the verification is required to initial and date all calibration activities. Any defects that occur during verification are also documented along with the corrective action applied and a demonstration of return to control. Annual service and calibration reports and certificates retained on file with QA staff.

Temperature control is documented in logbooks assigned to the equipment being monitored. A verified (see 7.1) thermometer is assigned to each individual item. Measurements are recorded along with date and initials of the individual conducting the measurement on a daily or as used basis. Corrective action, if required, is also documented including the demonstration of return to control.

Initial traceability of chemical standards and reagents is documented via a vendorsupplied certificate (see also 7.3) that includes lot number and expiration date information. Solutions prepared using the vendor supplied chemical standard are documented in logbooks assigned to specific analytical processes. Alternatively, documentation may be entered into the electronic standards and reagent tracking log The documentation includes links to the vendors lot number, an internal lot number, dates of preparation, and the preparer's initials. Standards received without certificate of analysis can not be used for calibration or calibration verification and are rejected.







Supervisors conduct regular reviews of logbooks, which are verified using a word rev'd", signature and date. QA Staff monitors the process and documents it in the same manner.



8.0 TEST PROCEDURES, METHOD REFERENCES, AND REGULATORY PROGRAMS

Requirements: The laboratory must use client specified or regulatory agency approved methods for the analysis of environmental samples. The laboratory maintains a list of active methods, which specifies the type of analysis performed, and cross-references the methods to applicable environmental regulation. Routine procedures used by the laboratory for the execution of a method must be documented in a standard operating procedure. Method performance and sensitivity must be demonstrated annually where required. Defined procedures for the use of method sensitivity for data reporting purposes must be established by the Director of Quality Assurance and used consistently for all data reporting purposes.

Method Selection. Accutest employs methods for environmental sample analysis that are consistent with the client's application, which are appropriate and applicable to the project objectives. Accutest informs the client if the method proposed is inappropriate or outdated and suggests alternative approaches.

Accutest employs documented, validated regulatory methods in the absence of a client specification and informs the client of the method selected. These methods are available to the client and other parties as determined by the client. Documented and validated in-house methods may be applied if they are appropriate to the project. The client is informed of the method selection.

8.2 <u>Method Validation</u>. Standard methods from regulatory sources are primarily used for all analysis. Standard methods do not require validation by the laboratory. Non-standard, in-house methods are validated prior to use. Validation is also performed for standard methods applied outside their intended scope of use. Validation is dependent upon the method application and may include analysis of quality control samples to develop precision and accuracy information for the intended use. A final method validation report is generated, which includes all data in the validation study. A statement of adequacy and/or equivalency is included in the report. A copy of the report is archived in the quality assurance directory of the company server.

Non-standard methods are validated prior to use. This includes the validation of modified standard methods to demonstrate comparability with existing methods. Demonstrations and validations are performed and documented prior to incorporating technological enhancements and non-standard methods into existing laboratory methods used for general applications. The demonstration includes method specific requirements for assuring that significant performance differences do not occur when the enhancement is incorporated into the method. Validation is dependent upon method application and may include the analysis of quality control samples to develop precision and accuracy information for intended use.

The study procedures and specifications for demonstrating validation include comparable method sensitivity, calibration response, method precision, method accuracy and field sample consistency for several classes of analytical methods are



detailed in this document. These procedures and specifications may vary depending upon the method and the modification.

- 8.3 <u>Standard Operating Procedures</u>. Standard operating procedures (SOP) are prepared for routine methods executed by the laboratory and processes related to sample or data handling. The procedures describe the process steps in sufficient detail to enable an individual, who is unfamiliar with the procedure to execute it successfully. SOPs are reviewed annually and edited if necessary. SOPs can be edited on a more frequent basis if systematic errors dictate a need for process change or the originating regulatory agency promulgates a new version of the method. Procedural modifications are indicated using a revision number. SOPs are available for client review at the Accutest facility upon request.
- 8.4 Method Detection Limit Determination and verification. Annual method detection limit (MDL) studies are performed as appropriate for routine methods used in the laboratory. MDL studies are also performed when there is a change to the method that affects how the method is performed or when an instrumentation change that impacts sensitivity occurs. The procedure used for determining MDLs is described in 40 CFR, Part 136, Appendix B. Studies are performed for each method on water, soil and air matrices for every instrument that is used to perform the method. MDLs are established at the instrument level. The highest MDL of the pooled instrument data is used to establish a laboratory MDL. MDLs are experimentally verified through the analysis of spiked quality control samples at 2-3 times the concentration of the experimental MDL, or 1-4 times for multicomponent methods. The verification is performed on every instrument used to perform the analysis. The quality assurance staff manages the annual MDL determination process and is responsible for retaining MDL data on file. Approved MDLs are appended to the LIMS and used for data reporting purposes. MDL values are used as DL values for DOD projects and verification spiking concentrations are listed ad LOD values.

Methods certified under DOD ELAP requirements must undergo verification procedure on quarterly basis – see DOD QSM 4.2, Gray Box D-13.

8.5 <u>Method Reporting Limit.</u> The method reporting limit is established at the lowest concentration calibration standard in the calibration curve. The low calibration standard is selected by department managers as the lowest concentration standard that can be used while continuing to meet the calibration linearity criteria of the method being used. The validity of the Method Reporting Limits is confirmed via analysis of a spiked quality control sample at 1 – 2x Method reporting limit concentration. RL values are referred to as LOQ for DOD projects.

By definition, detected analytes at concentrations below the low calibration standard cannot be accurately quantitated and must be qualified accordingly.

Methods certified under DOD ELAP requirements must undergo verification procedure on quarterly basis – see DOD QSM 4.2, Gray Box D-14.



Reporting of Quantitative Data. Analytical data for all methods is reported without qualification to the reporting limit established for each method. Data may be reported to MDL depending upon the client's requirements provided that all qualitative identification criteria for the parameter have been satisfied. All parameters reported at concentrations between the reporting limit and MDL are qualified as an estimated concentration.

Measured concentrations of detected analytes that exceed the upper limit of the calibration range are either diluted into the range and reanalyzed or qualified as an estimated value. The only exception to this applies to ICP and ICP/MS analysis, which can be reported to the upper limit of the experimentally determined linear range without qualification.

- 8.7 <u>Estimated Uncertainty.</u> A statement of the estimated uncertainty of an analytical measurement accompanies the test result when required. Estimated uncertainty is derived from the performance limits established for spiked samples of similar matrices. The degree of uncertainty is derived from the negative or positive bias for spiked samples accompanying a specific parameter. When the uncertainty estimate is applied to a measured value, the possible quantitative range for that specific parameter at that measured concentration is defined. Well recognized regulatory methods that specify values for the major sources of uncertainty and specify the data reporting format do not require a further estimate of uncertainty.
- 8.8 Precision and Accuracy Studies. Annual precision and accuracy (P&A) studies, which demonstrate the laboratories ability to generate acceptable date, are performed for all routine methods used in the laboratory. The procedure used for generating P&A data is referenced in the majority of the regulatory methodology in use. The procedure requires quadruplicate analysis of a sample spiked with target analytes at a concentration in the working range of the method. This data may be compiled from a series of existing blank spikes or laboratory control samples. Accuracy (percent recovery) of the replicate analysis is averaged and compared to established method performance limits. Values within method limits indicate an acceptable performance demonstration. (See also Sec 4, Training, Demonstration of capability)
- **Method Sources, References and Update Mechanism.** The Quality Assurance Staff maintains a list of active methods used for the analysis of samples. This list includes valid method references such as EPA, American Society of Testing and Materials (ASTM) or Standard Methods designations and the current version and version date.

Updated versions of approved reference methodology are placed into use as changes occur. The Quality Assurance Director informs operations management of changes in method versions as they occur. The operations management staff selects an implementation date. The operations staff is responsible for completing all method requirements prior to the implementation date. This includes modification to SOPs, completion of MDL and precision and accuracy studies and staff training. Documentation of these activities is provided to the QA staff who retains this information on file. The updated method is placed into service on the implementation date and the old version is de-activated.



Multiple versions of selected methods may remain in use to satisfy client specific needs. In these situations, the default method version becomes the most recent version. Client specific needs are communicated to the laboratory staff using method specific analytical codes method, which clearly depict the version to be used. The old method version is maintained as an active method until the specified client no longer requires the use of the older version.

Accutest will not use methodology that represents significant departures from the reference method unless specifically directed by the client. In cases where clients direct the laboratory to use a method modification that represents a significant departure from the reference method, the request will be documented in the project file. The LQSM lists active methods used for the analysis of samples in Table 8.1. This list includes valid method references from sources such as USEPA, ASTM or Standard Methods designations and the current version and version date.

8.10 <u>Analytical Capabilities</u>. Appendix II provides a detailed listing of the methodology employed for the analysis of test samples.



9.0 SAMPLE MANAGEMENT, LOGIN, CUSTODY, STORAGE AND DISPOSAL

Requirement: A system to ensure that client supplied product is adequately evaluated, acknowledged, and secured upon delivery to the laboratory must be practiced by the laboratory. The system must assure that chain of custody is maintained and that sample receipt conditions and preservation status are documented and communicated to the client and internal staff. The login procedure must assign, document, and map the specifications for the analysis of each unique sample to assure that the requested analysis is performed on the correct sample and enables the sample to be tracked throughout the laboratory analytical cycle. The system must include procedures for reconciling defects in sample condition or client provided data, which occur at sample arrival. The system must specify the procedures for proper sample storage, transfer to the laboratory, and disposal after analysis. The system must be documented in a standard operating procedure.

Order Receipt and Entry. New orders are initiated and processed by the client services group (See Chapter 14, Procedures for Executing Client Specifications). The new order procedure includes mechanisms for providing sampling containers to clients. These containers must meet the size, cleanliness, and preservation specifications for the analysis to be performed.

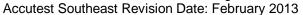
For new orders, the project manager prepares a bottle request form, which is submitted to sample management department. This form provides critical project details to the sample management staff, which are used to prepare and assemble the sample bottles for shipment to the client prior to sampling.

The bottle order is assembled using bottles that meet USEPA specifications for contaminant-free sample containers. Accutest-SE checks all sample containers for cleanliness. Data are reviewed by both the analyst and sample management technician. Results of bottle analyses are retained for minimum of 5 years.

All preservative solutions are prepared in the laboratory and are checked to assure that they are free of contamination from analytes of interest before being released for use. Sample management department retains a copy of the documentation of in-house contamination checks.

Reagent water for trip and field blanks is poured into appropriately labeled containers. Sample bottleware is labeled with durable labels printed on waterproof printing medium with indelible laser or heat transfer printer ink. All bottles are packed into ice chests with blank chain of custody forms and the original bottle order form. Completed bottle orders are delivered to clients using Accutest couriers or commercial carriers for use in field sample collection.

9.2 <u>Sample Receipt and Custody</u>. Samples are delivered to the laboratory using a variety of mechanisms including Accutest couriers, commercial shippers, and client self-delivery. Documented procedures are followed for arriving samples to assure that





custody and integrity are maintained and that handling and preservation requirements are documented and continued.

Sample custody documentation is initiated when the individual collecting the sample collects field samples. Custody documentation includes all information necessary to provide an unambiguous record of sample collection, sample identification, and sample collection chronology. Initial custody documentation employs either Accutest or client generated custody forms.

Accutest generates a Sample Receipt Confirmation form in situations where the individuals who collected the sample did not generate custody documentation in the field. Accutest SE Project Manager then contacts the client for the CoC information to be faxed or e-mailed from the client to the lab.

Accutest defines sample custody as follows:

- The sample is in the actual custody or possession of the assigned responsible person,
- The sample is in a secure area.

The Accutest facility is defined as a secure facility. Perimeter security has been established, which limits access to authorized individuals only. Visitors enter the facility through the building lobby and must register with the receptionist prior to entering controlled areas. While in the facility, visitors must be accompanied by their hosts at all times. After hours, building access is controlled using a computerized pass-key reader system. This system limits building access to individuals with a preassigned authorization status. After hours visitors are not authorized to be in the building. Clients delivering samples after hours must make advanced arrangements through client services and sample management to assure that staff is available to take delivery and maintain custody.

Upon arrival at Accutest, the sample custodian reviews the chain of custody and generates Sample Receipt Confirmation form for the samples received to verify that the information on the form corresponds with the samples delivered. This includes verification that all listed samples are present and properly labeled, checks to verify that samples were transported and received at the required temperature, verification that the sample was received in proper containers, verification that sufficient volume is available to conduct the requested analysis, and a check of individual sample containers to verify test specific preservation requirements including the absence of headspace for volatile compound analysis.

9.3 Sample conditions and other observations are documented on the Sample Receipt Confirmation form by the sample custodian prior to completing acceptance of custody. The sample custodian accepts sample custody upon verification that the custody document is correct. Discrepancies or non-compliant situations are documented, flagged and communicated to the Accutest project manager, who contacts the client



for resolution. The resolution is documented and communicated to sample management for execution.

9.4 <u>Laboratory preservation of Improperly preserved field samples.</u> Accutest extends every effort to preserve samples which were received without proper field preservation.

Field/Equipment negative controls also receive the same amount of preservation as incorrectly preserved samples, and record made in the preservation logbook.

9.5 <u>Sample Tracking Via Status Change.</u> An automated, electronic LIMS procedure records sample exchange transactions between departments and changes in analytical status. This system tracks all preparation, analytical, and data reporting procedures to which a sample is subjected while in the possession of the laboratory. Each individual receiving samples must acknowledge the change in custody and operational status in the LIMS. This step is required to maintain an accurate electronic record of sample status, dates of analytical activity, and custody throughout the laboratory.

Sample tracking is initiated at login where all chronological information related to sample collection dates and holding times are entered into the LIMS. This information is entered on an individual sample basis

Sample Acceptance Policy. Incoming samples must satisfy Accutest's sample acceptance criteria before being logged into the system. Sample acceptance is based on the premise that clients have exercised proper protocols for sample collection. This includes sufficient volume, proper chemical preservation, temperature preservation, sample container sealing and labeling, and appropriate shipping container packing.

The sample management staff will make every attempt to preserve improperly preserved samples upon arrival. However, if preservation is not possible, the samples may be refused unless the client authorizes analysis. No samples will be accepted if holding times have been exceeded or will be exceeded before analysis can take place unless the client authorizes analysis.

Sample acceptance criteria include proper custody and sample labeling documentation. Proper custody documentation includes an entry for all physical samples delivered to the laboratory with an identification code that matches the sample bottle and a date and signature of the individual who collected the sample and delivered them to the laboratory. Labeling is done using durable waterproof labels printed with indelible heat-transfer ink.

Accutest reserves the right to refuse any sample which in its sole and absolute discretion and judgement is hazardous, toxic and poses or may pose a health, safety or environmental risk during handling or processing. The company will not accept samples for analysis using methodology that is not performed by the laboratory or for methods that lab does not hold valid accreditation unless arrangements have been made to have the analysis conducted by a qualified subcontractor.



9.7 Assignment of Unique Sample Identification Codes. Unique identification codes must be assigned to each sample bottle to assure traceability and unambiguously identify the tests to be performed in the laboratory.

The sample identification coding process begins with the assignment of a unique alphanumeric job number. A job is defined as a group of samples received on the same day, from a specific client pertaining to a specific project. A job may consist of groups of samples received over multi-day period. The first character of the job number is an alphacharacter that identifies the laboratory facility. The next characters are numeric and sequence by one number with each new job.

Unique sample numbers are assigned to each bottle collected as a discrete entity from a designated sample point. This number begins with the job number and incorporates a second series of numbers beginning at one and continuing chronologically for each point of collection. The test to be performed is clearly identified on the bottle label.

Alpha suffixes may be added to the sample number to identify special designations such as subcontracted tests, in-house QC checks, or re-logs. Multiple sample bottles for a specific analysis are labeled Bottle 1, Bottle 2, etc.

9.8 <u>Subcontracted Analysis</u>. Subcontract laboratories are employed to perform analysis not performed by Accutest. The quality assurance staff evaluates subcontract laboratories to assure their quality processes meet the standards of the environmental laboratory industry prior to engagement. Throughout the subcontract process, Accutest follows established procedures to assure that sample custody is maintained and the data produced by the subcontractor meets established quality criteria.

Accutest network laboratories are considered primary subcontractors.

Subcontracting Procedure. Subcontracting procedures are initiated through several mechanisms, which originate with sample management. Samples for analysis by a subcontractor are logged into the Accutest system using regular login procedures. If subcontract parameters are part of the project or sample management has received subcontracting instructions for a specific project, a copy of the chain of custody is given to the appropriate project manager with the subcontracted parameters highlighted. This procedure triggers the subcontract process at the project management level. The Sample Management supervisor contacts an approved subcontractor to place the subcontract order. Subcontract chain of custody is processed in Sample Management Department and copy is filed with the original CoC. Sample management signs the subcontract chain of custody and ships the sample(s) to the subcontractor. The subcontract COC is filed with the original COC and the request for subcontract. Copies are distributed to the login department, the project manager, and sample management.

Client is verbally notified by Project Manager of the requirement to subcontract to the outside laboratory as soon as need Is identified by the Accutest staff. Client notification





must be verified in writing, i.e. by e-mail. Client notification may take place during the initial project set-up, or at the time of sample receipt and login.

Subcontractor data packages are reviewed by the QA Staff to assess completeness and quality compliance. If completeness defects are detected, the subcontractor is asked to immediately upgrade the data package. If data quality defects are detected, the package is forwarded to the QA staff for further review. The QA staff will pursue a corrective action solution before releasing data to the client.

Approved subcontract data is entered into the laboratory information management system (LIMS) if possible and incorporated into the final report. All subcontract data is footnoted to provide the client with a clear indication of its source. Copies of original subcontract data are always included in the data report whether in hardcopy or PDF file, depending on the data submission requirements.

Subcontract Laboratory Evaluation. The QA staff evaluates subcontract laboratories prior to engagement. As a minimum, the subcontract laboratory must provide Accutest with proof of a valid certification to perform the requested analysis for the venue where they were collected, QC criteria summary (LOD/LOQ, LCS, MS/MSD, %RPD, etc.), copy of the most recent regulatory agency audit report, and a copy of the laboratory's Summary of Qualifications (SOQ). Other beneficial materials are QSM, copies of SOPs used for the subcontracted analysis, a copy of the most recent performance evaluation study for the subcontracted parameter, and copies of the most recent third party accreditor's audit report.

Certification verification must be submitted to Accutest annually. If possible, the QA staff may conduct a site visit to the laboratory to inspect the quality system. Accutest Laboratories Southeast assumes the responsibility for the performance of all subcontractors who have successfully demonstrated their qualifications. When selecting a subcontractor for analysis not performed by Accutest, assure qualifications of the subcontractor through local QA officer.

Qualification process of a subcontract laboratory may be bypassed if the primary client directs Accutest to employ a specific subcontractor

Subcontract Laboratory Database. Accutest Laboratories Inc. maintains centralized database of preferred contractors in order to optimize sample handling and data submission process, as well as obtain competitive priced services of uniform quality throughout the network. Individual Accutest laboratories are assigned "Center of Expertise" status according to unique capabilities.

Sample Storage. Following sample custody transfer, samples are assigned to various refrigerated storage areas by the sample management staff depending upon the test to be performed and the matrix of the samples. The location (refrigerator and shelf) of each sample is entered into sample location database on the line corresponding to each sample number. Samples remain in storage until the laboratory technician retrieves them into the laboratory for analysis.



Samples for volatile organics analysis are placed in storage in designated refrigerators by the sample management staff and immediately transferred to the organics group control. Sample custody is transferred to the VOC department staff. These samples are segregated according to matrix to limit opportunities for cross contamination to occur.

Organics staff is authorized to retrieve samples from these storage areas for analysis. When analysis is complete, the samples are placed back into storage.

9.10 <u>Sample Login</u>. Following sample custody transfer to the laboratory, the documentation that describes the clients analytical requirements are delivered to the sample login group for coding and entry to the Laboratory Information management System (LIMS). This process translates all information related to collection time, turnaround time, sample analysis, and deliverables into a code which enables client requirements to be electronically distributed to the various departments within the laboratory for scheduling and execution.

The technical staff is alerted to client or project specific requirements through the use of a unique project code that is electronically attached to the job during login. The unique project code directs the technical staff to controlled specifications documents detailing the unique requirements.

9.11 <u>Sample Retrieval for Analysis</u>. It is a responsibility of individual analyst to retrieve samples for analysis. Sample Management employs a program to facilitate sample placement and retrieval. Sample is traced around the laboratory using Status feature of LIMS.

After sample analysis has been completed, the analyst places the sample back into the storage and updates sample status.

9.12 <u>Sample Disposal</u>. Accutest retains all samples under proper storage for a minimum of 30 days following completion of the analysis report. Longer storage periods are accommodated on a client specific basis if required. Samples may also be returned to the client for disposal.

Accutest disposes of all laboratory wastes following the requirements of the Resource Conservation and Recovery Act (RCRA). The Company has obtained and maintains a waste generator identification number, FLR00001263309002 (FLR designates State of Florida).

Sample management generates a sample disposal dump sheet from the LIMS tracking system each week, which lists all samples whose holding period has expired. Data from each sample is compared to the hazardous waste criteria established by the Florida Department of Environmental Protection (FDEP).

Samples containing constituents at concentrations above the criteria are labeled as hazardous and segregated into the following waste categories for disposal as follows:



Chlorinated Waste (Closed Top Steel Drum)- Methylene Chloride

Non-Chlorinated Waste (Closed Top Steel Drum)- Hexane, Methanol, and mixed solvents

Sodium Sulfate/Used Charcoal (Open Top Steel Drum)- Charcoal and paper filters used in the filtering of samples.

Hazardous Flammable Vials (Open Top Polypropylene Drum)- Methylene Chloride, Hexane.

Hazardous Aqueous waste (Closed Top Polypropylene Drum)- High Odor Samples, Lachat Waste.

Non Hazardous Soil (Open Top Steel Drum)- Soils.

Hazardous Solid Waste- (Open Top Steel Drum).

Non-Aqueous/Oil Samples- (Closed Top Steel Drum)

Difference between Open and Closed type of drums is whether it is possible to remove entire lid or just threaded stopper. Drums are closed at all times while in storage.

Non-hazardous aqueous samples are neutralized and collected in HDPP 500 Gal holding tank to be removed by waste company.

Non-hazardous solids are drummed and disposed of by contract waste company. Sample bottles are disposed of as recyclable waste in order to crush the bottles and destroy the labels. VOC vials are crushed on site using PRODEVA glass crusher. Supernatant liquid is siphoned off into the HDPP holding tank and solid residue drummed separately.

Laboratory wastes are collected by waste stream in designated areas throughout the laboratory. Waste streams are consolidated twice a week by the waste custodian and transferred to stream specific drums for disposal through a permitted waste management contractor. Filled, consolidated drums are tested for hazardous characteristics and scheduled for removal from the facility for appropriate disposal based on the laboratory data.



10.0 LABORATORY INSTRUMENTATION AND MEASUREMENT STANDARDS

Requirement: Procedures, which assure that instrumentation is performing to a predetermined operational standard prior to the analysis of any samples, must be established by the laboratory. In general, these procedures will follow the regulatory agency requirements established in promulgated methodology. The instrumentation selected to perform specified analysis is capable of providing the method-specified uncertainty and sufficient sensitivity of measurement needed. These procedures must be documented and incorporated into the standard operating procedures for the method being executed. ALSE Equipment List attached as Appendix III.

- Mass Tuning Mass Spectrometers. The mass spectrometer tune and sensitivity must be monitored to assure that the instrument is assigning masses and mass abundances correctly and that the instrument has sufficient sensitivity to detect compounds at low concentrations. This is accomplished by analyzing a specific mass tuning compound at a fixed concentration. If the sensitivity is insufficient to detect the tuning compound, corrective action must be performed prior to the analysis of standards or samples. If the mass assignments or mass abundances do not meet criteria, corrective action must be performed prior to the analysis of standards or samples.
- 10.2 <u>Wavelength Verification Spectrophotometers</u>. Spectrophotometer detectors are checked on a regular schedule to verify proper response to the wavelength of light needed for the test in use. If the detector response does not meet specifications, corrective action (detector adjustment or replacement) is performed prior to the analysis of standards or samples.
- 10.3 <u>Inter-element Interference Checks (Metals)</u>. Inductively Coupled Plasma Emission Spectrophotometers (ICP) are subject to a variety of spectral interferences, which can be minimized or eliminated by applying interfering element correction factors and background correction points. Interfering element correction factors are checked on a specified frequency through the analysis of check samples containing high levels of interfering elements. Analysis of single element interferent solutions is also conducted at a specified frequency.

If the check indicates that the method criteria has not been achieved for any element in the check standard, the analysis is halted and data from the affected samples are not reported. Sample analysis is resumed after corrective action has been performed and the correction factors have been re-calculated.

New interfering element correction factors are calculated and applied whenever the checks indicate that the correction factors are no longer meeting criteria. At a minimum, correction factors are replaced once a year.



10.4 <u>Calibration and Calibration Verification</u>. Many tests require calibration using a series of reference standards to establish the concentration range for performing quantitative analysis. Method specific procedures for calibration are followed prior to any sample analysis.

Calibration is performed using a linear or quadratic regression calculation or calibration factors calculated from the curve. The calibration must meet method specific criteria for linearity or precision. If the criteria are not achieved, corrective action (instrument maintenance or re-calibration) is performed. The instrument must be successfully calibrated before analysis of samples can be conducted.

Initial calibration for metals analysis performed using inductively coupled plasma (ICP) employs the use of two standards and a calibration blank to establish linearity. The calibration blank contains all reagents that are placed into the calibration standard with the exception of the target elements. Valid calibration blanks must not contain any target elements.

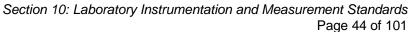
Initial calibrations must be initially verified using a single concentration calibration standard from a second source (i.e. separate lot or different provider). The continuing validity of an existing calibration must be regularly verified using a single concentration calibration standard. The response to the standard must meet pre-established criteria that indicate the initial calibration curve remains valid. If the criteria are not achieved corrective action (re-calibration) is performed before any additional samples may be analyzed.

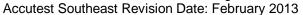
10.5 <u>Linear Range Verification and Calibration</u> Linear range verification is performed for all ICP instrumentation and select General Chemistry methods. The regulatory program or analytical method specifies the verification frequency. A series of calibration standards are analyzed over a broad concentration range. The data from these analyses are used to determine the valid analytical range for the instrument.

Some methods or analytical programs require a low concentration calibration check to verify that instrument is sufficient to detect target elements at the reporting limit. The analytical method or regulatory program defines the criteria used to evaluate the low concentration calibration check. If the low calibration check fails criteria, corrective action is performed and verified through reanalysis of the low concentration calibration check before continuing with the field sample analysis.

In accordance with TNI standards minimum number of calibration points in the absence of method-specific requirements is two calibration points and a blank.

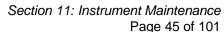
10.6 <u>Retention Time Verification (GC/HPLC/IC)</u>. Chromatographic retention time windows are developed for all analysis performed using gas chromatographs with conventional detectors. An initial experimental study is performed, which establishes the width of the retention window for each compound. The retention time range of the window defines the time ranges for elution of specified target analytes on the primary and







confirmation columns. Retention time windows are established upon initial calibration, applying the retention time range from the initial study to each target compound. Retention times are regularly confirmed through the analysis of an authentic standard during calibration verification. If the target analytes do not elute within the defined range during calibration verification, the instrument must be recalibrated and new windows defined. New studies are performed when major changes, such as column replacement are made to the chromatographic system.



Accutest Southeast Revision Date: February 2013



11.0 INSTRUMENT MAINTENANCE

Requirement. Procedures must be established for equipment maintenance. The procedure may include a maintenance schedule if required or documentation of daily maintenance related activities. All instrument maintenance activities must be documented in instrument specific logbooks. All equipment out of service (both analytical and auxiliary) must be clearly marked "Out of Order".

- 11.1 Routine, Daily Maintenance. Routine, daily maintenance is required on an instrument specific basis. It is performed each time the instrument is used. Daily maintenance traditionally includes activities to insure a continuation of good analytical performance. In some cases, they include performance checks that indicate whether non-routine maintenance is required. If the performance check indicates a need for higher level maintenance, the equipment is taken out of service until maintenance is performed. Analysis cannot be continued until the performance checks meet established criteria. Document return to control. Daily maintenance is the responsibility of the individual assigned to the instrument used for the analysis he is performing.
- 11.2 Non-routine Maintenance. Non-routine maintenance is reserved for catastrophic occurrences such as instrument failure. The need for non-routine maintenance is indicated by failures in general operating systems that result in an inability to conduct required performance checks or calibration. Equipment in this category are taken out of service and repaired before attempting further analysis. Analysis cannot continue until the instrument meets all performance check criteria and is capable of being calibrated. Section supervisors are responsible for identifying non-routine maintenance episodes and initiating repair activities to bring the equipment on-line. This may include initiating telephone calls to maintenance contractors if necessary. They are also responsible for documenting all details related to the occurrence and the repair.
- 11.3 <u>Scheduled Maintenance</u>. Modern laboratory instrumentation rarely requires regular preventative maintenance. Where required, the equipment is placed on a schedule, which dictates when maintenance is required. Examples include annual balance calibration by an independent provider and optical alignment of the ICP. Section supervisors are responsible for initiating scheduled maintenance on equipment that requires scheduled preventative attention. Scheduled maintenance is documented using routine documentation practices.
- Maintenance Documentation. Routine and non-routine maintenance activities are documented in logbooks assigned to instruments and equipment used for analytical measurements. The logbooks contain preprinted forms, which specify the maintenance activities required with each use. Accutest Laboratories Southeast has adopted a problem action follow-up format to conduct instrument maintenance. The analyst or supervisor who performs or initiates the maintenance activity is required to check the activity upon its completion, verify complete statement of return to normal conditions and initial the form. Non-routine maintenance (i.e. repairs, upgrades, etc.) is documented as well either electronically via e-mail from the service provider or receipt attached to the maintenance log.



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12.0 QUALITY CONTROL PARAMETERS, PROCEDURES, AND CORRECTIVE ACTION

Requirement: All procedures used for test methods must incorporate quality control parameters to monitor elements that are critical to method performance. Each quality parameter includes acceptance criteria that have been established by regulatory agencies for the methods in use. Criteria may also be established through client dictates or through the accumulation and statistical evaluation of internal performance data. Data obtained from these parameters must be evaluated by the analyst, and compared to established method criteria. If the criteria are not achieved, the procedures must specify corrective action and conformation of control before proceeding with sample analysis. QC parameters, procedures, and corrective action must be documented within the standard operating procedures for each method. In the absence of client specific objectives the laboratory must define qualitative objectives for completeness and representativeness of data.

Procedure. Bench analysts are responsible for methodological quality control and sample specific quality control. Each method specifies the control parameters to be employed for the method in use and the specific procedures for incorporating them into the analysis. These control parameters are analyzed and evaluated with every designated sample group (batch).

The data from each parameter provides the analyst with critical decision making information on method performance. The information is used to determine if corrective action is needed to bring the method or the analysis of a specific sample into compliance. These evaluations are conducted throughout the course of the analysis. Each parameter being indicative of a critical control feature. Failure of a methodological control parameter is indicative of either instrument or batch failure. Failure of a sample control parameter is indicative of control difficulties with a specific sample or samples.

Sample Batch. All samples analyzed in the laboratory are assigned to a designated sample batch, which contains all required quality control samples and a defined maximum number of field samples that are prepared and/or analyzed over a defined time period. The maximum number of investigative and field QC samples in the batch is 20. Accutest has incorporated the NELAP batching policy as the sample-batching standard. This policy incorporates the requirement for blanks and spiked blanks as a time based function as defined by NELAP. The typical batch contains a blank, laboratory control sample (LCS or spiked blank), matrix spike and matrix spike duplicate. Batch documentation includes lot specifications for all reagents and standards used during preparation of the batch.

Methodological Control Parameters and Corrective Action. Prior to the analysis of field sample the analyst must determine that the method is functioning properly. Specific control parameters indicate whether critical processes meet specified requirements before continuing with the analysis. Method specific control parameters must meet criteria before sample analysis can be conducted. Each of these



parameters is related to processes that are under the control of the laboratory and can be adjusted if out of control.

Method Blank. A method blank is analyzed during the analysis of any field sample. The method blank is defined as a sample. It contains the same standards (internal standards, surrogates, matrix modifiers, etc.) and reagents that are added to the field sample during analysis, with the exception of the sample itself. If the method blank contains target analyte(s) at concentrations that exceed method or client requirements (typically defined as 1/2 RL concentrations), the source of contamination is eliminated before proceeding with sample analysis. Systematic contamination is documented for corrective action and resolved following the established corrective action procedures. In specific cases, contamination detected in the method blank may be acceptable if the concentrations do not exceed regulatory limits or client defined reporting limits.

Laboratory Control Samples (LCS or Spiked Blanks). A laboratory control sample (spiked blank or commercially prepared performance evaluation sample) is analyzed along with field samples to demonstrate that the method accuracy is within acceptable limits. These spike solutions are derived from different sources than the solutions used for method calibration. The performance limits are derived from published method specifications or from statistical controls generated from laboratory method performance data. Spiked blanks are blank matrices (reagent water or clean sand) spiked with the targeted parameters and analyzed using the same method used for samples. Accuracy data is compared to laboratory experimentally derived limits to determine if the method is in control. Laboratory control samples (LCS) are commercially prepared spiked samples in an inert material. Performance criteria for recovery of spiked analytes is pre-established by the commercial entity preparing the sample. This sample is analyzed in the laboratory as an external reference.

Accuracy data is compared to the applicable performance limits. If the spike accuracy exceeds the performance limits, corrective action, as specified in the SOP for the method is performed and verified before continuing with a field sample analysis. In some cases, decisions are made to continue with sample analysis if performance limits are exceeded; provided the unacceptable result has no negative impact on the sample data.

Marginal exceedance (ME) values are calculated for methods containing more than eleven (11) targeted analytes. The ME is calculated as \pm 4 standard deviations about the mean. MEs are considered for multi-analyte methods because of the increased likelihood of LCS failure as the number of analytes in the method increase. The number of allowable MEs is based on the number of target analytes in the method. Analytes that regularly fall into the ME category are treated as systematic problems, which are resolved using established trend monitoring and corrective action procedures. Marginal Exceedances are not applied to parameters that are detected in field samples. Routine corrective action is initiated for all cases where LCS spike accuracy criteria is beyond the established control limits and the parameter is detected in field samples corresponding to the unacceptable LCS.



Blanks and spikes are routinely evaluated before samples are analyzed. However, in situations where sample analysis is performed using an autosampler, they may be evaluated after sample analysis has occurred. If the blanks and spikes do not meet criteria, sample analysis is repeated.

Proficiency Testing. Performance Evaluation (Proficiency Testing) samples (PEs, PTs) are single or double blind samples spiked with know amount of analytes on interest and introduced to the laboratory to assess method performance. PEs may be introduced as double blinds submitted by commercial clients, single or double blinds from regulatory agencies, or internal blinds submitted by the QA group.

A minimum of two single blind studies must be performed each year for every parameter in aqueous and solid matrices for each field of proficiency testing (FOPT) for which the laboratory maintains accreditation. Proficiency Testing samples must be purchased as blinds from an accredited vendor. Data from these studies are provided to the laboratory by the vendor and reported to accrediting agencies. If unsatisfactory performance is noted, corrective action is performed to identify and eliminate any sources of error. A new PT must be analyzed to demonstrate continuing proficiency.

PE samples performed for accrediting agencies or clients, which do not meet performance specifications, require a written summary that documents the corrective action investigation, findings, and corrective action implementation.

Single or double blind PT samples are employed for self-evaluation purposes. Data from these analyses are compared to established performance limits. If the data does not meet performance specifications, the system is evaluated for sources of acute or systematic error. If required, corrective action is performed and verified before initiating or continuing sample analysis.

Trend Analysis for Control Parameters. Accuracy data for selected spiked parameters from the laboratory control sample (LCS) is statistically evaluated daily for trends. Data from selected LCS parameters and surrogates are pooled on a method, matrix, and instrument basis. This data is evaluated by comparison to existing control and warning limits. Trend analysis is performed automatically as follows:

- Any point outside the control limit
- Any three consecutive points between the warning and control limits
- Any eight consecutive points on the same side of the mean
- Any six consecutive points increasing or decreasing

The results of the trend analysis are printed for supervisory evaluation prior to sample analysis. Trends that indicate the potential loss of statistical control are further evaluated to determine the impact on data quality and to determine if corrective action is necessary. If corrective action is indicated, the supervisor informs the analysts of



the corrective actions to be performed. Return to control is demonstrated before analysis resumes.

12.3 <u>Sample Control Parameters and Corrective Action</u>. The analysis of samples can be initiated following a successful demonstration that the method is operating within established controls. Additional controls are incorporated into the analysis of each sample to determine if the method is functioning within established specifications for each individual sample. Sample QC data is evaluated and compared to established performance criteria. If the criteria are not achieved the method or the SOP specifies the corrective action required to continue sample analysis. In many cases, failure to meet QC criteria is a function of sample matrix and cannot be remedied. Each parameter is designed to provide quality feedback on a defined aspect of the sampling and analysis episode.

Duplicates. Duplicate sample analysis is used to measure analytical precision. This can also be equated to laboratory precision for homogenous samples. Precision criteria are method dependent. If precision criteria are not achieved, corrective action or additional action may be required. Recommended action must be completed before sample data can be reported.

Laboratory Control Duplicate, Spikes & Spiked Duplicates. Spikes and spiked duplicates are used to measure analytical precision and accuracy for the sample matrix selected. Precision and accuracy criteria are method dependent. If precision and accuracy criteria are not achieved, corrective action or additional action may be required. Recommended action must be completed before sample data can be reported.

Serial Dilution (Metals). Serial dilutions of metals samples are analyzed to determine if analytical matrix effects may have impacted the reported data. If the value of the serially diluted samples does not agree with the undiluted value within a method-specified range, the sample matrix may be causing interference, which may lead to either a high or low bias. If the serial dilution criterion is not achieved, it must be flagged to indicate possible bias from matrix effects. Accutest-SE uses this procedure as opposed to post-digestion spike unless contractual obligations absolutely require latter

Post Digestion Spikes. Digested samples are spiked and analyzed to determine if matrix interferences are creating biases in the results. It may also be used to determine potential interferences per client's specification. Spike concentration is determined as per analytical method. No action is necessary if the post digestion spike is outside of the method criteria, unless a preparation problem is suspected with the spike, in which case the post digestion spike should remade and reanalyzed.

Surrogate Spikes (Organics). Surrogate spikes are organic compounds that are similar in behavior to the target analytes but unlikely to be found in nature. They are added to all quality control and field samples to measure method performance for each individual sample. Surrogate accuracy limits are derived from published method



specifications or by statistical evaluation of laboratory generated surrogate accuracy data. Accuracy data is compared to the applicable performance limits. If the surrogate accuracy exceeds performance limits, corrective action, as specified in the method or SOP is performed before sample data can be reported.

Internal Standards (Organic Methods). Internal standards are retention time and instrument response markers added to every sample to be used as references for quantitation. Their response is compared to reference standards and used to evaluate instrument sensitivity on a sample specific basis. Internal standard retention time is also compared to reference standards to assure that target analytes are capable of being located by their individual relative retention time.

If internal standard response criteria are not achieved, corrective action or additional action may be required. The recommended action must be completed before sample data can be reported.

If the internal standard retention time criteria are not achieved corrective action or additional action may be required. This may include re-calibration and re-analysis. Additional action must be completed before sample data is reported.

Internal Standards (ICP Metals). Internal standards are used on ICP instruments to compensate for variations in response caused by differences in sample matrices. This adjustment is performed automatically during sample analysis. The internal standard response of replicated sample analysis is monitored to detect potential analytical problems. If analytical problems are suspected, then the field samples are reanalyzed.

12.4 <u>Laboratory Derived Quality Control Criteria.</u> Control criteria for in-house methods and client specific modifications that exceed the scope of published methodology are defined and documented prior to the use of the method. The Quality Assurance staff identifies the responsibility for control criteria needs. Control parameters and criteria, based on best technical judgement are established using input provided by the operations staff. These control parameters and criteria are documented and incorporated into the method.

The laboratory derived criteria are evaluated for technical soundness on spiked samples prior to the use of the method on field samples. The technical evaluation is documented and archived by the Quality Assurance staff.

When sufficient data form the laboratory developed control parameter is accumulated, the data is statistically processed and the experimentally derived control limits are incorporated into the method.

12.5 <u>Bench Review & Corrective Action</u>. The bench chemists are responsible for all QC parameters. Before proceeding with sample analysis, they are required to successfully meet all instrumental QC criteria. They have the authority to perform any necessary corrective action before proceeding with sample analysis. Their authority



includes the responsibility for assuring that departures from documented policies and procedures do not occur.

The bench chemists are also responsible for all sample QC parameters. If the sample QC criteria are not achieved, they are authorized and required to perform the method specified corrective action before reporting sample data.

<u>Data Qualifiers</u>. An alpha character coding system is employed for defining use limitations for reported data. These limitations are applied to analytical data by the analyst to clarify the usefulness of the reported data for data user. Accutest Laboratories Southeast qualifies data in accordance with program-specific requirements, such as State of Florida DEP, AFCEE, etc., and these qualifiers are hard-coded in the LIMS on project level. Definitions of common qualifiers could be found at the bottom of the sample report form.

12.6 <u>QA Monitoring</u>. The QA staff prior to client release conducts a spot review of completed data packages. This review includes an examination of QC data for compliance and trends indicative of systematic difficulties. If non-conformances are detected, the QA staff places an immediate stop on the release of the data and initiates corrective action to rectify the situation. The data package is released when the package becomes compliant with all quality requirements.

If the review reveals trends indicative of systematic problems, QA initiates an investigation to determine the cause. If process defects are detected, a corrective action is implemented and monitored for effectiveness.

Performance Limits. The Technical Director is responsible for compilation and maintenance of all precision and accuracy data used for performance limits. Quality control data for all test methods are accumulated and stored in the laboratory information management system (LIMS). Parameter specific QC data is extracted annually and statically processed to eliminate outliers and develop laboratory specific warning limits and confidence limits. The new limits are reviewed and approved by the supervisory staff prior to their use for data assessment. The new limits are used to evaluate QC data for compliance with method requirements for a period of one year. Laboratory generated limits appear on all data reports unless method specifies hard-coded limits (mostly General Chemistry and Metals)

12.7 <u>Data Package Review</u>. Accutest employs multiple levels of data review to assure that reported data has satisfied all quality control criteria and that client specifications and requirements have been met. Production departments have developed data review procedures which must be conducted before data is released to the client.

Analytical Review. The analyst conducts the primary review of all data. This review begins with a check of all instrument and method quality control and progresses through sample quality control concluding with a check to assure that the client's requirements have been executed. Analyst checks focuses on a review of qualitative



determinations and checks of precision and accuracy data to verify that existing laboratory criteria have been achieved. Checks at this level may include comparisons with project specific criteria if applicable. The analyst has the authority and responsibility to perform corrective action for any out-of-control parameter or nonconformance at this stage of review.

Secondary data reviews are performed at the peer level by analysts who have met the qualification criteria for the method in use. Qualification requirements include a valid demonstration of capability and demonstrated understanding of the method SOP. Section supervisors may perform secondary review in-lieu of a peer review Secondary review is performed on 100% of the data produced by their department. It includes a check of all manual calculations; an accuracy check of manually transcribed data from bench sheets to the LIMS, a check of all method and instrument QC criteria, baseline manipulations (if applicable) and a comparison of the data package to client specified requirements. Also included are checks to assure the appropriate methodology was applied and that all anomalous information was properly flagged for communication in the case narrative. Supervisors have the authority to reject data and initiate reanalysis, corrective action, or reprocessing.

All laboratory data requiring manual entry into LIMS system is double-checked by the analysts performing initial data entry and the section supervisor. Verification of supervisory review is indicated on the raw data summary by the supervisor's initials and date.

Electronic data that is manually edited at the bench by the primary analysts is automatically flagged by the instrument data system indicating an override by the analyst. All manual overrides must be verified and approved by a supervisor who initials and dates all manual changes.

Hard copies of manually integrated chromatographic peaks are printed that clearly depict the manually drawn baseline. The hard copy is reviewed and approved by the reviewer (initialed and dated) and included in the data package of all full tier reports or the archived batch records of commercial report packages.

Electronic data that has been committed to the LIMS can only be edited by a manager or supervisor. These edits may be required if needs for corrections are indicated during the final review. An audit record for all electronic changes in the LIMS is automatically appended to the record.

The group manager performs a tertiary review on a spot check basis. This review includes an evaluation of QC data against acceptance criteria and a check of the data package contents to assure that all analytical requirements and specifications were executed.

Report Generation Review. The report generation group reviews all data and supporting information delivered by the laboratory for completeness and compliance



with client specifications. Missing deliverables are identified and obtained from the laboratory. The group also reviews the completed package to verify that the delivered product complies with all client specifications. Non-analytical defects are corrected before the package is sent to the client.

Project Management/Quality Assurance Review. Spot-check data package reviews are performed by the project manager. Project management reviews focus on project specifications. If the project manager identifies defects in the product prior to release, he initiates immediate corrective action to rectify the situation.

The QA Staff reviews approximately 10% of the data produced. The QA review focuses on all elements of the deliverable including the client's specifications and requirements, analytical quality control, sample custody documentation and sample identification. QA reviews at this step in the production process are geared towards systematic process defects, which require procedural changes to effect a corrective action. However, if defects are identified that can be corrected prior to data release, the QA staff returns the package to the laboratory for corrective action. QA data review cannot be used in lieu of a peer level review or a supervisory review.

Data Reporting. Analytical data is released to clients following secondary departmental review. Data release at this stage of the process is limited to electronic information, which is released to clients through a secure, encrypted, password protected, Internet connection.

Hard copy support data is compiled by the report generation group and assembled into the final report. The report is sent to the client following reviews by report generation, and spot-check by QA staff.

All data reports include specified information, which is required to identify the report and its contents. This information includes a title, name and address of the laboratory, a unique report number, total number of pages in the report, clients name and address, analytical method identification, arriving sample condition, sample and analysis dates, test results with units of measurement, authorized signature of data release, statement of applicability, report reproduction restrictions and TNI requirements certification. Subcontracted data is clearly identified.

In the event of report revision date of the revision, nature of revision and identity of the person revising the report must be clearly stated in the body of the report. Depending on the level of the deliverables it could be either stated in the Case Narrative or Case Narrative generated specifically for this purpose. Case Narrative must state "supercedes all previous reports".

12.8 Electronic Data Reduction. Raw data from sample analysis is entered into the laboratory information management system (LIMS) using automated processes or manual entry. Final data processing is performed by the LIMS using procedures developed by the Company.



All LIMS programs and internally developed software (including Excel spreadsheets) are tested and validated prior to use to assure that they consistently produce correct results. Validation testing is performed by the Information Technology Staff. The testing procedures are documented in an SOP. Programs are not approved for use until they have demonstrated that they are capable of performing the required calculations.

- **Representativeness**. Data representativeness is based on the premise that qualitative and quantitative information developed for field samples is characteristic of the sample that was collected by the client and analyzed in the laboratory. The laboratory objective for representativeness defines data as representative if the criteria for all quality parameters associated with the analysis of the sample are achieved.
- **12.10** <u>Comparability</u>. Analytical data is defined as comparable when data from a sample set analyzed by the laboratory is representatively equivalent to other sample sets analyzed separately regardless of the analytical logistics. The laboratory will achieve 100% comparability for all sample data which meets the criteria for the quality parameters associated with its analysis using the method requested by the client.



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13.0 CORRECTIVE ACTION SYSTEM

<u>Requirement</u>. The laboratory must have polices and procedures for correcting defective processes, systematic errors, and quality defects, which enables the staff to systematically improve product quality. The system must include procedures for communicating items requiring corrective action, corrective action tracking procedures, corrective action documentation, monitoring of effectiveness, and reports to management. The system must be documented in a standard operating procedure.

Procedure. Corrective action is the step that follows the identification of a process defect. The type of defect determines the level of documentation, communication, and training necessary to prevent re-occurrence of the defect or non-conformance.

Routine Corrective Action. Routine corrective action is defined as the procedures used to return out of control analytical systems back to control. This level of corrective action applies to all analytical quality control parameters or analytical system specifications.

Bench analysts have full responsibility and authority for performing routine corrective action. The resolution of defects at this level does not require a procedural change or staff re-training. The analyst is free to continue work once corrective action is complete and the analytical system has been returned to control. Documentation of routine corrective action is limited to bench logbook or maintenance logbook comment.

Process Changes. Corrective actions in this category require procedural modifications. They may be the result of systematic defects identified during audits, the investigation of client inquiries, failed proficiency tests, product defects identified during data review, or method updates. Resolution of defects of this magnitude requires formal identification of the defect, development and documentation of a corrective action plan, and staff training to communicate the procedural change.

Technical Corrective Action. Technical corrective action encompasses routine corrective action performed by bench analysts for out of control systems and corrective actions performed for data produced using out of control systems. Technical corrective action for routine situations is conducted using the procedures detailed above.

Non-routine corrective actions apply to situations where the bench analysts failed to perform routine corrective action before continuing analysis. Supervisors and Department Managers perform corrective action in these situations. Documentation of all non-routine corrective actions is performed using the corrective action system.

Sample re-analysis is conducted if sufficient sample and holding time remain to repeat the analysis using an in-control system. If insufficient sample or holding time remains, the data is processed and qualifiers applied that describe the out of control situation. The occurrence is further documented in the case narrative and in the corrective



action response. The corrective action must include provisions for retraining the analysts who failed to perform routine corrective action.

13.2 Documentation & Communication. Routine corrective actions are documented as part of the analytical record. Notations are made in the comments section of the analytical chronicle or data sheet detailing the nonconformance. Continuation of the analysis indicates that return to control was successful.

Corrective actions for process changes are documented, tracked and monitored for effectiveness. Corrective actions may be initiated by any supervisor or senior staff member by completing the corrective action form in Corrective Action database

The corrective action database is an Access application. The initiator generates the corrective action investigation form, which is documented, tracked, distributed to responsible parties and archived through the application. The application assigns a tracking number initiation data and due date to each corrective action initiated and copies the corrective action form to the corrective action database. The application also distributes an E-mail message containing the form to the responsible parties for resolution.

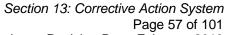
Corrective Action system employs Deficiency – Root Cause – Immediate Fix – Corrective action approach, further divided into categories of Analytical Error, Omission Error, Random Error, Systemic Error and Training Issue.

The responsible party develops and implements the procedural change. Existing documentation such as SOPs are edited to reflect the change. The affected staff is informed of the procedural change through a formal training session. The training is documented and copies are placed into individual training files. The corrective action form is completed and closed in CA database.

Initial and completed corrective action forms are maintained in the Corrective Action directory. This information is archived daily. Copies of training records describing corrective actions are appended to the involved individuals training files.

Monitoring. The QA Staff monitors the implemented corrective action until it is evident that the corrective action has been effective and the systematic deficiency has been eliminated. The corrective action database is updated by QA to reflect closure of the corrective action. The QA staff also assigns an error code to the corrective action for classification of the type of errors being committed.

If QA determines that the corrective action procedure has not effectively remedied the deficiency, the process continues with a re-initiation of the corrective action. Corrective action continues until the defective process is eliminated. If another procedural change is required, it is treated as a new corrective action, which is documented and monitored using established procedures.





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Client Notification. Defective processes, systematic errors, and quality defects, detected during routine audits may have negative impacts on data quality. In some cases, data that has been released to clients may be affected. If defective data has been released for use, Accutest will notify the affected clients of the defect and provide specific details regarding the magnitude of the impact to their data.





14.0 PROCEDURES FOR EXECUTING CLIENT SPECIFICATIONS

Requirement. Systems must be established for evaluating and processing client specifications for routine and non-routine analytical services. The systems must enable the client services staff to identify, evaluate, and document the requested specifications to determine if adequate resources are available to perform the analysis. The system must include procedures for communicating the specifications to the laboratory staff for execution and procedures for verifying the specifications have been executed.

14.1 *Client Specific Requirements.* The project manager is the primary contact for clients requesting laboratory services. Client specifications are communicated using several The primary source of information is the client's quality assurance mechanisms. project plan (QAPP) which details analytical and quality control specifications for the project. In the absence of a QAPP, projects specifications can also be communicated using contracts, letters of authorization, or letters of agreement, which may be limited to a brief discussion of the analytical requirements and the terms and conditions for the work. These documents may also include pricing information, liabilities, scope of work, in addition to the analytical requirements. QAPPs include detailed analytical requirements and data quality objectives, which supersede those found in the referenced methods. This information is essential to successful project completion.

Laboratory also reviews its Accreditation status to evaluate whether it is possible to accept proposed project. Discrepancies must be resolved before the work commences.

The client services staff provides additional assistance to clients who are unsure of the specifications they need to execute the sampling and analysis requirements of their project. They provide additional support to clients who require assistance in results interpretation as needed, provided they possess the expertise required to render an opinion.

The project manager is responsibility for obtaining project documents, which specify the analytical requirements. Following project management review, copies are distributed to the QA staff and the appropriate departmental managers for review and comment. The original QAPP is numbered with a document control number and filed in a secure location.

Requirements for Non-Standard Analytical Specifications. Client requirements 14.2 that specify departures from documented policies, procedures, or standard specifications must be submitted to Accutest in writing. These requirements are reviewed and approved by the technical staff before the project is accepted. Once accepted, the non-standard requirements become analytical specifications, which follow the routine procedure for communicating client specifications. Departures from documented policies, procedures, or standard specifications that do not follow this procedure are not permitted.



Exception Policy: With respect to the quality system, incoming non-conforming product refers to received samples that do not meet requirements of custody documentation, are improperly packaged or stored or are contaminated. An internal non-conformance refers to a problem, caused internally due to improper handling of samples, improper sampling methods, and equipment malfunction or data management errors. The individual who identifies the incoming non-conformance is responsible for notifying the project manager. The project manager resolves the issue with the client. The individual who recognizes an internal non-conformance is responsible for initiating corrective action

Departures from standard practices, policies and specifications are reviewed and approved by Technical Director, QA Officer and by Project Manager of the project affected.

Corrective & Preventative Action: Once a quality problem has been identified, the analytical or review process stops, until the reason is identified. Primary responsibility for identifying the cause of the problem rests with the instrument operator. Other staff may be called on to assist in reaching the root cause. The problem prevention tracking system, using Corrective Action Tracking Records, provides a method to track systemic problems until resolved/removed. The QA Officer is responsible for the record management with respect to the disposition of problems.

Deviations that do not limit themselves to a single department and/or client are cited on Corrective Action Record. This may include but not limited to: sample arrival outside of EPA specified holding time, analysis completion outside of EPA specified holding time (with explanation of the reason), inconsistencies between chain of custody and cooler contents, including labeling errors, improper preservation, etc.

Deviations from analytical methods' SOP's are reported by the Analyst to the Section Leader. Single occurrences warrant completion of Corrective Action Tracking record, repetitive occurrences may indicate that either an additional training session is in order, or the SOP does not reflect proper laboratory practice. Training session is conducted by the Technical Director or by QA Officer. In case where SOP does not reflect current laboratory practice, SOP review and correction process may be initiated.

14.3 <u>Evaluation of Resources.</u> A resource evaluation is completed prior to accepting projects submitted by clients. The evaluation is initiated by the client services staff receives project requirements (usually in the form of QAPjP) and distributes these requirements to the laboratory departments affected. The specifications are evaluated by the department managers from a scheduling and hardware resources perspective. The project is not accepted unless the department managers have the necessary resources to execute the project according to client specifications.



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14.4 <u>Documentation</u>. New projects are initiated using a project set up form, which is completed prior to the start of the project. This form details all of the information needed to correctly enter the specifications for each client sample into the laboratory information management system (LIMS, see example). The form includes data reporting requirements, billing information, data turnaround times, QA level, state of origin, and comments for detailing project specific requirements. The project manager is responsible for obtaining this information from the client and completing the form prior to sample arrival and login.

Sample receipt triggers project creation and the login process. The information on the set-up form is entered into the LIMS immediately prior to logging in the first sample. The set up form may be accompanied by a quotation, which details the analytical product codes and sample matrices. These details are also entered into the LIMS during login.

Special information is distributed to the laboratory supervisors and login department in electronic or hardcopy format upon project setup. All project specific information is retained by the project manager in a secure file. The project manager maintains a personal telephone log, which details conversations with the client regarding the project.

14.5 <u>Communication</u>. A pre-project meeting is held between client services and the operations managers to discuss the specifications described in the QAPjP and/or related documents. Project logistics are discussed and finalized and procedures are developed to assure proper execution of the client's analytical specifications and requirements. Questions, raised in the review meeting, are discussed with the client for resolution. Exceptions to any requirements, if accepted by the client, are documented and incorporated into the QAPjP or project documentation records.

Non-standard specifications for individual clients are documented in the LIMS at the client account level. Once entered into the LIMS, these specifications become memorialized for all projects related to the client account. Upon sample arrival, these specifications are accessed through a terminal or printed as a hard copy and stored in a binder for individuals who require access to the specification. Specifications that are not entered into the LIMS are prohibited unless documented in an interdepartmental memo, which clearly identifies the project, client and effective duration of the specification.

14.6 <u>Operational Execution</u>. A work schedule is prepared for each analytical department on a daily basis. Analytical specifications from recently arrived samples have now been entered into the LIMS database. The database is sorted by analytical due date and holding time, into product specific groups. Samples are scheduled for analysis by due date and holding time. The completed schedule, which is now defined as a work list, is printed. The list contains the client requested product codes and specifications required for the selected sample(s). Special requirements are communicated to the analyst using the comments section or relayed through verbal instructions provided by



the supervisor. The bench analyst assumes full responsibility for performing the analysis according to the specifications printed on the work sheet.

14.7 <u>Verification</u>. Prior to the release of data to the client, laboratory section managers and the report generation staff review the report and compare the completed product to the client specifications documentation to assure that all requirements have been met. Project managers perform a spot check of projects with unique requirements to assure that the work was executed according to specifications.



15.0 CLIENT COMPLAINT RESOLUTION PROCEDURE

Requirement. A system for managing and reconciling client complaints must be implemented in the laboratory. The system must include procedures for documenting client complaints and communicating the complaint to the appropriate department for resolution. The system must also include a quality assurance evaluation to determine if the complaint is related to systematic defects requiring process changes.

Procedure. Client complaints are communicated to client services representatives, quality assurance staff, or senior management staff for resolution. The individual receiving the complaint retains the responsibility for documentation and communicating the nature of the complaint to the responsible department(s) for resolution. The responsible party addresses the complaint. The resolution is communicated to quality assurance (QA) and the originator for communication to the client. QA reviews the complaint and resolution to determine if systematic defects exist. If systematic defects are present, QA works with the responsible party to develop a corrective action that eliminates the defect.

<u>Documentation</u>. Client's complaints are documented by the client service representative receiving the complaint. A record of the telephone conversation is maintained by client services. Client service staff enters the complaint into Data Challenge database or Client Complaint database, depending on the nature of complaint. These databases are cross-linked with corrective action database – see sec. 13. Complaint is communicated to the production departments concerned via auto e-mail. The complaint resolution is documented in the database by the responsible party and resultant e-mail returned to the originator. QA staff is copied on the correspondence.

- 15.2 <u>Corrective Action</u>. Responses to Data Challenges/Client Complaints are required from the responsible party. At a minimum, the response addresses the query and provides an explanation to the complaint. Corrective action may focus on the single issue expressed in the complaint. Corrective action may include job case narrative generation, reprocessing of data, editing of the initial report, and re-issue to the client. If the QA review indicates a systematic error, process modification is required. The defective process at the root of the complaint is changed. SOPs are either created or modified to reflect the change. The party responsible for the process implements process changes.
- **QA Monitoring.** Process changes, implemented to resolve systematic defects, are monitored for effectiveness by QA. If monitoring indicates that the process change has not resolved the defect, QA works with the department management to develop and implement an effective process. If monitoring indicates that the defect has been resolved, monitoring is slowly discontinued. Continued monitoring is incorporated as an element of the annual system audit and annual Management Report (see 18.8).





16.0 CONTROL OF NONCONFORMING PRODUCT

Requirement: Policies and procedures have been developed and implemented that describe the procedures employed by the laboratory when any aspect of sample analysis or data reporting do not conform to established procedures or client specifications. These procedures include steps to ensure that process defects are corrected and affected work is evaluated to assess its impact to the client.

Procedure. Nonconforming product is identified through multiple channels, such as second level analytical data review, routine internal review and audit practices, external auditing or through client inquiry. Responsibility and authority for the management of the non-conforming product directly defined by a nature of a nonconformance. For example, non-conformances resulting from internal and external reviews are evaluated and managed by QA Staff. Corrective Action items are issued and followed to completion and verification that defect is prevented from reoccurring. Non-conformances stemming from client inquiry are managed by Project Management staff with QA staff oversight.

Data associated with out-of compliance QC are evaluated by bench personnel and section supervisors. The analyst has the authority and responsibility to perform corrective action for any out-of-control parameter or nonconformance at this stage of

If non-conformances are detected, the QA staff places an immediate stop on the release of the data and initiates corrective action to rectify the situation

Non-conformances and their significance are communicated in case narrative and sample report footnotes. Case narrative comments and sample repot footnotes must state the impact on data quality.

Corrective Action. The outcome of the evaluation dictates the course of action. The type of defect determines the level of documentation, communication, and training necessary to prevent re-occurrence of the defect or non-conformance This may include at a minimum client notification, but may also include corrective action. Immediate corrective action is performed using the SOP-specified procedures. However, additional action may be required including cessation of analysis and withholding and/or recalling data reports. If the evaluation indicates that nonconforming data may have been issued to clients, the client is immediately notified and data may be recalled following the procedures specified in respective SOPs. If work has been stopped because of a nonconformance, the Laboratory Director is the only individual authorized to direct a resumption of analysis.

Nonconformances caused by systematic process defects require retraining of the personnel involved as an element of the corrective action solution. Routine corrective actions are documented as part of the analytical record.



17.0 CONFIDENTIALITY PROTECTION PROCEDURES

Requirements: Policies and procedures are required to protect client data from release to unauthorized parties or accidental release of database information through accidental electronic transmission or illegal intrusion. These policies must be communicated to clients and staff. Electronic systems must be regularly evaluated for effectiveness.

17.1 Client Anonymity. Information related to the Company's clients is granted to employees on a "need to know" basis. An individual's position within the organization defines his "need to know". Individuals with "need to know" status are given password access to systems that contain client identity information and access to documents and document storage areas containing client reports and information. Access to client information by individuals outside of the Company is limited to the client and individuals authorized by the client.

Individuals outside of the Company may obtain client information through subpoena issued by a court of valid jurisdiction. Clients are informed when subpoenas are received ordering the release of their information.

- 17.2 **Documents.** Access to client documents is restricted to employees in need to know positions. Copies of all client reports are stored in secure archive with restricted Reports and report copies are distributed to individuals who have been access. authorized by the client to receive them. Documents are not released to third parties without verbally expressed or written permission from the client.
- 17.3 Confidential Business Information (CBI). Operational documents including SOPs, Quality Manuals, personnel information, internal operations statistics, and laboratory audit reports are considered confidential business information. Strict controls are placed on the release of this information to outside parties.

Release of CBI to outside parties or organizations may be authorized upon execution of a confidentiality agreement between Accutest and the receiving organization or individual. CBI information release is authorized for third party auditors and commercial clients in electronic mode as Adobe Acrobat .PDF format only. See also Sec. 6.5.

17.4 Electronic Data.

Database Intrusion. Direct database entry is authorized for employees of Accutest only on a need to know basis. Entry to the database is restricted through a user specific multiple password entry system. Direct access to the database outside of the facility is possible through a VPN connection. A unique password is required for access to the local area network. A second unique password is required to gain access to the database. The staff receives read or write level authorization on a hierarchical privilege basis.



Internet Access. Access to client information is through an HTTP Web application only. It does not contain a mechanism that allows direct access to the database. Clients can gain access to their data only using a series of Accutest assigned accounts, and client specific passwords. The viewable data, which is encrypted during transmission, consists of an extraction of database information only.

Client Accessibility. Accessibility to client data delivered via electronic means follows strict protocols to insure confidentiality. Clients accessing electronic data are assigned a company account. The account profile, which is established by the MIS staff, grants explicit access to explicit information pertaining to the clients project activity. Passwords are assigned on an individual basis within a client account. These accounts can be activated or deactivated by the MIS staff only.

- 17.5 <u>Information Requests</u>. Client specific data or information is not released to third parties without verbally expressed or written permission from the client. Written permission is required from third parties, who contact the Company directly for the release of information. Verbal requests will be honored only if they are received directly from the client. These requests must be documented in a record of communication maintained by authorized recipient.
- 17.6 <u>Transfer of Records</u>. Archived data, which has previously been reported and transmitted to clients, is the exclusive property of Accutest Laboratories. In the event of a cessation of business activities due to business failure or sale, The Company's legal staff will be directed to arrange for the final disposition of archived data.

The final disposition of archived data will be accomplished using the approach detailed in the following sequence:

- 1. All data will be transferred to the new owners for the duration of the required archive period as a condition of sale.
- If the new owners will not accept the data or the business has failed, letters will be sent to clients listed on the most recent active account roster offering them the option to obtain specific reports (identified by Accutest Job Number) at their own expense.
- A letter will be sent to the TNI accrediting authority with organizational jurisdiction over the company offering them the option to obtain all unclaimed reports at their own expense.
- 4. All remaining archived data will be recycled using the most expedient means possible.



18.0 QUALITY AUDITS AND SYSTEM REVIEWS

<u>Requirement</u>: The quality assurance group will conduct regularly scheduled audits of the laboratory to assess compliance with quality system requirements, technical requirements of applied methodology, and adherence to documentation procedures. The information gathered during these audits will be used to provide feedback to senior management and perform corrective action where needed for quality improvement purposes.

- Quality Systems Review. Quality system audits are performed annually by the Quality Assurance Director for the Company President. In this audit, the laboratory is evaluated for compliance with the Laboratory Quality Systems Manual (LQSM) and the quality system standards of the National Environmental Laboratory Accreditation Conference. Findings, which indicate non-compliance or deviation from the LQSM, are flagged for corrective action. Corrective actions require either a return to compliance or a plan change to reflect an improved quality process. The QA Officer is responsible for making and documenting changes to the LQSM. These changes are reviewed by the Laboratory Director and Technical Director prior to the approval of the revised system.
- Quality System Audits. Quality system audits are conducted to evaluate the effectiveness and laboratory compliance with individual quality system elements. These audits are conducted on an established schedule. Audit findings are documented and communicated to the management staff and entered into the corrective action system for resolution. If necessary, retraining is conducted to assure complete understanding of the system requirements.
- 18.3 <u>Technical Compliance Audits</u>. Technical compliance audits are performed throughout the year following the established schedule. Selected analytical procedures are evaluated for compliance with standard operating procedures (SOPs) and method requirements. If non-conformances exist, the published method serves as the standard for compliance. SOPs are edited for compliance if the document does not reflect method requirements. Analysts are trained to the new requirements and the process is monitored by quality assurance. Analysts are retrained in method procedures if an evaluation of bench practices indicates non-compliance with SOP requirements.
- 18.4 <u>Documentation Audits</u>. Documentation audits are conducted periodically. This audit includes a check of measurement processes that require manual documentation and non-analytical logbook review. It also includes checks of data archiving systems and a search to find and remove any inactive versions of SOPs that may still be present in the laboratory and being accessed by the analysts. Non-conformances are corrected on the spot. Procedural modifications are implemented if the evaluation indicates a systematic defect.
- **18.5** <u>Corrective Action Monitoring</u>. Defects or non-conformances that are identified during client or internal audits are shared with management and entered into CA



database for attention by the responsible party. Audit findings are corrected through process modifications and/or retraining. Once a corrective action has been designed and implemented, it is monitored for compliance on a regular basis by the QA staff. Monitoring of the corrective action continues until satisfactory implementation has been verified.

- **Preventive Action.** Laboratory systems or processes, which may be faulty and pose the potential for nonconformances, errors, confusing reports or difficulties establishing traceability may be identified during internal audits. These items are highlighted for systematic change using the corrective action system and managed to resolution using appropriate procedures for corrective action.
- 18.7 <u>Client Notification.</u> Defective processes, systematic errors, and quality defects detected during routine audits may have negative impact on data quality. In some cases, data that has been released to the client may be affected. If defective data has been released for use, Accutest will immediately notify the affected clients of the defect and provide specific details regarding the magnitude of the impact to their data.
- **18.8** <u>Management Reports</u>. Formal reports of all audit activities are prepared for the management staff. These reports are prepared annually. The report details the status of the Quality System

The formal report also addresses the following topics:

- the suitability of policies and procedures;
- reports from managerial and supervisory personnel;
- the outcome of recent internal audits;
- corrective and preventive actions;
- assessments by external bodies;
- the results of interlaboratory comparisons or proficiency tests;
- changes in the volume and type of the work;
- customer feedback;
- complaints;
- recommendations for improvement;
- other relevant factors, such as quality control activities, resources, and staff training.





19.0 HEALTH AND SAFETY

Requirement. The company operates a formal health and safety program that complies with the requirements of the Occupational Health and Safety Administration. The program consists of key policies and practices that are essential to safe laboratory operation. All employees are required to receive training on the program elements. Job specific training is conducted to assure safe practices for specific tasks. All employees are required to participate in the program, receive initial and annual training, and comply with the program requirements. All plan and program requirements are detailed in the Health and Safety Program Manual.

19.1 **Policy.** Accutest Laboratories will provide a safe and healthy working environment for its employees and clients while protecting the public and preserving the Company's The company will comply with all applicable government assets and property. regulations pertaining to safety and health in the laboratory and the workplace.

The objective of the Accutest Health and Safety Program is to promote safe work practices that minimize the occurrence of injuries and illness to the staff through proper health and safety training, correct laboratory technique application and the use of engineering controls.

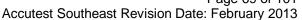
19.2 **Responsibilities.** The Health and Safety Program assists managers, supervisors and non-supervisory employees in control of hazards and risks to minimize the potential for employee and client injuries, damage to client's property and damage or destruction to Accutest's facility.

The Health and Safety Officer is responsible for implementing the Program's elements and updating its contents as necessary. He also conducts periodic audits to monitor compliance and assess the program's effectiveness and is also responsible for creating and administering safety training for all new and existing employees.

The employee is responsible for following all safety rules established for their protection, the protection of others and the proper use of protective devices provided by the Company. The employee is also expected to comply with the requirements of the program at all times. Department Managers and Supervisors are responsible for ensuring the requirements of the Safety Program are practiced daily. The Company President retains the ultimate responsibility for the program design and implementation.

19.3 **Program Elements.** The Accutest Health and Safety Program consists of key program elements that compliment the company's health and safety objective. These elements form the essence of the health and safety policy and assure that the objectives of the program are achieved.

Safety Education and Training and Communication. Training is conducted to increase the staff's awareness of laboratory hazards and their knowledge of the safety





practices and procedures required to protect them from those hazards. It is also used to communicate general safety procedures required for safe operation in a chemical laboratory.

Initial health and safety training for new employees is conducted during orientation. The training focuses on the Accutest Safety and Health Program and includes specific training for the hazards that may be associated with the employees' duties. Training is also conducted for all program elements focusing on general, acceptable, laboratory safety procedures. Targeted training is conducted to address hazards or safety procedures that are specific to individual employee's work assignments. All training activities are documented and archived in individual training folders. A health and safety training inventory is maintained in the training database.

Accutest Laboratories Southeast maintains personnel trained in HAZWOPER, DOT and HazMat operations, as well as respirator certified.

Safety Officer. The safety officer provides the employees with an opportunity to express their views and concerns on safety issues in an environment where those concerns will be addressed to ensure that the interests of the company and the well being of the employee are protected. Safety Officer is entrusted with elevating the level of safety awareness among their peers.

Hazard Identification and Communication. The hazard communication program enables employees to readily identify laboratory hazards and the procedures to protect themselves from those hazards. This program complies with OSHA's Hazard Communication Standard, Title 29 Code of Federal Regulations 1910.1200 that requires the company to adopt and adhere to the following key elements:

- Material Safety Data Sheets (MSDS) and/or Safety Data Sheets (SDS) must be available to any employee wishing to view them,
- The Company must maintain a Hazardous Chemicals Inventory (by location), which is updated on an annual basis,
- Containers are properly labeled,
- All employees must be provided with annual Personal Protection, Hazard Communication and Right to Know training,

Chemical Hygiene Plan. The Chemical Hygiene Plan complies with the requirements of the Occupational Safety and Health Administration's Occupational Exposure to Hazardous Chemicals in the Laboratory Standard, 29 CFR 1910.1450. This plan establishes procedures, identifies safety equipment, personal protective equipment, and work practices that protect employees from the potential health hazards presented by hazardous chemicals in the laboratory if properly used and/or applied.





Emergency Action & Evacuation Plan. The Emergency Action and Evacuation Plan details the procedures used to protect and safeguard Accutest's employees and property during emergencies. Emergencies are defined as fires or explosions, gas leaks, building collapse, hazardous material spills, emergencies that immediately threaten life and health, bomb threats and natural disasters such as floods, hurricanes or tornadoes. The plan identifies and assigns responsibility for executing specific roles in situations requiring emergency action.

Lockout/Tagout Plan. Lockout/tagout procedures have been established to assure that laboratory employees and outside contractors take steps to render equipment inoperable and/or safe before conducting maintenance activities. The plan details the procedures for conducting maintenance on equipment that has the potential to unexpectedly energize, start up, or release energy or can be operated unexpectedly or accidentally resulting in serious injury to employees. The plan ensures that employees performing maintenance render the equipment safe through lock out or tag out procedures.

Personal Protection Policy. Policies have been implemented which detail the personal protection requirements for employees. The policy includes specifications regarding engineering controls, personal protective equipment (PPE), hazardous waste, chemical exposures, working with chemicals and safe work practices. Safety requirements specific to processes or equipment are reviewed with the department supervisor or the Health and Safety Officer before beginning operations.

Emergency Preparedness Plan. This plan identifies the actions to be taken by Accutest Laboratory's staff in the event of terrorism or terrorist actions, to ensure the safety of the employees and the facility. The plan describes the building security actions coinciding with the "Alert Condition", designated by the Department of Homeland Security.



Appendix I

Glossary of Terms



GLOSSARY OF TERMS

Acceptance Criteria: specified limits placed on characteristics of an item, process, or service defined in requirement documents.

Accreditation: the process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one.

Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

Analyst: the designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

Analytical Uncertainty: A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis.

Audit: a systematic evaluation to determine the conformance to quantitative and qualitative specifications of some operational function or activity.

Batch: environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one to 20 environmental samples of the same quality-system matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

Blank: a sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results.

Blind Sample: a sub-sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.

Case Narrative: a statement of non-conformances associated with particular data report



Calibration: to determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter, instrument, or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements.

Calibration Curve: the mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response.

Calibration Method: a defined technical procedure for performing a calibration.

Calibration Standard: a substance or reference material used to calibrate an instrument.

Certified Reference Material (CRM): a reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body.

Chain of Custody: an unbroken trail of accountability that ensures the physical security of samples and includes the signatures of all who handle the samples.

Clean Air Act: the enabling legislation in 42 U.S.C. 7401 *et seq.*, Public Law 91-604, 84 Stat. 1676 Pub. L. 95-95, 91 Stat., 685 and Pub. L. 95-190, 91 Stat., 1399, as amended, empowering EPA to promulgate air quality standards, monitor and to enforce them.

Comprehensive Environmental Response, Compensation and Liability Act (CERCLA/Superfund): the enabling legislation in 42 U.S.C. 9601-9675 *et seq.*, as amended by the Superfund Amendments and Reauthorization Act of 1986 (SARA), 42 U.S.C. 9601*et seq.*, to eliminate the health and environmental threats posed by hazardous waste sites.

Confirmation: verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to second column confirmation, alternate wavelength, derivatization, mass spectral interpretation, alternative detectors or, additional cleanup procedures.

Conformance: an affirmative indication or judgement that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements.

Corrective Action: the action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence.

Data Audit: a qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality (i.e., that they meet specified acceptance criteria).

Data Reduction: the process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form.



Demonstration of Capability: a procedure to establish the ability of the analyst to generate acceptable accuracy.

Document Control: the act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed.

Duplicate Analyses: the analyses or measurements of the variable of interest performed identically on two sub-samples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory.

Federal Water Pollution Control Act (Clean Water Act, CWA): the enabling legislation under 33 U.S.C. 1251 *et seq.*, Public Law 92-50086 Stat. 816, that empowers EPA to set discharge limitations, write discharge permits, monitor, and bring enforcement action for noncompliance.

Field of Testing: TNI's approach to accrediting laboratories by program, method and analyte. Laboratories requesting accreditation for a program-method-analyte combination or for an up-dated/improved method are required submit to only that portion of the accreditation process not previously addressed (see TNI, section 1.9ff).

Holding Times (Maximum Allowable Holding Times) the maximum times that samples may be held prior to analysis and still be considered valid or not compromised.

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample): a sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes from a source independent of the calibration standards or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

Matrix (or Quality System Matrix): the component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

Aqueous: any aqueous sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes surface water, groundwater, effluents, and TCLP or other extracts.

Drinking Water: any aqueous sample that has been designated a potable or potential potable water source. Saline/Estuarine: any aqueous sample from an ocean or estuary, or other saltwater source such as the Great Salt Lake. Non-aqueous Liquid: any organic liquid with <15% settleable solids.



Biological Tissue, Biota: any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Solids: includes soils, sediments, sludges and other matrices with >15% settleable solids.

Chemical Waste: a product or by-product of an industrial process that results in a matrix not previously defined.

Air: whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbent tube, impinger solution, filter, or other device.

Matrix Spike (spiked sample or fortified sample): a sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of Target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix Spike Duplicate (spiked sample or fortified sample duplicate): a second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

Method Blank: a sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest, which is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

Method Detection Limit: the minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

National Institute of Standards and Technology (NIST): an agency of the US Department of Commerce's Technology Administration that is working with EPA, States, TNI, and other public and commercial entities to establish a system under which private sector companies and interested States can be accredited by NIST to provide NIST-traceable proficiency testing (PT) to those laboratories testing drinking water and wastewater.

The NELAC institute (TNI): a voluntary organization of State and Federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories.

TNI Standards: the plan of procedures for consistently evaluating and documenting the ability of laboratories performing environmental measurements to meet nationally defined standards established by the The NELAC Institute.



Performance Audit: the routine comparison of independently obtained *qualitative* and *quantitative* measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory.

Precision: the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.

Preservation: refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample.

PT Fields of Testing: TNI's approach to offering proficiency testing by regulatory or environmental program, matrix type, and analyte.

Proficiency Testing: a means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source.

Proficiency Test Sample (PT): a sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria.

Quality Assurance: an integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

Quality Control: the overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users.

Quality Manual: a document stating the management policies, objectives, principles, oganizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.

Quality System: a structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC.

Quantitation Limits: the maximum or minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be quantified with the confidence level required by the data user.

Range: the difference between the minimum and the maximum of a set of values.



Raw Data: any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted.

Reagent Blank (method reagent blank or method blank): a sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps.

Reference Material: a material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.

Reference Method: a method of known and documented accuracy and precision issued by an organization recognized as competent to do so.

Reference Standard: a standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived.

Replicate Analyses: the measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval.

Requirement: denotes a mandatory specification; often designated by the term "shall".

Resource Conservation and Recovery Act (RCRA): the enabling legislation under 42 USC 321 *et seq.* (1976), that gives EPA the authority to control hazardous waste from the "Cradleto-grave", including its generation, transportation, treatment, storage, and disposal.

Safe Drinking Water Act (SDWA): the enabling legislation, 42 USC 300f *et seq.* (1974), (Public Law 93-523), that requires the EPA to protect the quality of drinking water in the U.S. by setting maximum allowable contaminant levels, monitoring, and enforcing violations.

Sample Duplicate: two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method including sampling and analysis.

Spike: a known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.



Standard: the document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of TNI and meets the approval requirements of TNI procedures and policies.

Toxic Substances Control Act (TSCA): the enabling legislation in 15 USC 2601 *et seq.*, (1976), that provides for testing, regulating, and screening all chemicals produced or imported into the United States for possible toxic effects prior to commercial manufacture.

Traceability: the property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons.

United States Environmental Protection Agency (EPA): the federal governmental agency with responsibility for protecting public health and safeguarding and improving the natural environment (i.e., the air, water, and land) upon which human life depends.

Validation: the process of substantiating specified performance criteria.

Verification: confirmation by examination and provision of evidence that specified requirements have been met.

NOTE: In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment. The result of verification leads to a decision either to restore in service, to perform adjustment, to repair, to downgrade, or to declare obsolete. In all cases, it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record.



Appendix II

Analytical Capabilities



TNI-Accredited Fields of Testing

Method Type	Method Number	Regulatory Program
Organics		
EDB and DBCP 1,4-Dioxane	EPA 504.1 EPA 522	Drinking Water Drinking Water
Metals		
ICP: General Cold Vapor Mercury	EPA 200.7, 1994 EPA 245.1, 1994	Drinking Water Drinking Water
Inorganic WetChem		
Perchlorate by Ion Chromatography	EPA 314.0	Drinking Water
Organics		
EDB and DBCP Volatile Organics Semi-Volatile Organics Semi-Volatile Organics Purgeable Aromatics Chlorinated Pesticides & PCBs Poly-Aromatic Hydrocarbons Nitroaromatics Explosives Explosives Chlorinated Herbicides Organophosphorus Pesticides Perchlorate Dissolved Gases Alcohols Gasoline Range Organics Diesel Range Organics Total Petroleum Hydrocarbons Tennessee EPH Tennessee GRO Wisconsin DRO	EPA 504, SW846 8011** EPA 624, SW846 8260B** EPA 625, SW846 8270D** SW846 8270D SIM** EPA 602, SW846 8021A** EPA 608, SW846 8081B**, 8082A** EPA 610, SW846 8310** SW846 8091** SW846 8330A**, 8332** SW846 8330B**, SW846 8151A** SW846 8141B** SW-846 6850 RSK SOP 147-175** SW846 8015C,D** SW846 8015C,D** SW846 8015C,D** TN-EPH** TN-GRO** WI-DRO**	Non-Potable Water
Petroleum Hydrocarbons Petroleum Hydrocarbons Volatile Petro. Hydrocarbons	Iowa OA-1** Iowa OA-2** Massachusetts VPH, 2004**	Non-Potable Water Non-Potable Water Non-Potable Water



Method Type	Method Number	Regulatory Program	
Extractable Petro. Hydrocarbons Total Petroleum Hydrocarbons Acrylamide	Massachusetts EPH, 1998** TX-1005** SW846 8316	Non-Potable Water Non-Potable Water Non-Potable Water	
Metals	GW040 0010	Non i otable water	
motars			
ICP: General – EPA WW	EPA 200.7, 1994; SW-846 6010C**	Non-Potable Water	
Cold Vapor Mercury – EPA WW	EPA 245.1, 1994; SW-846 7470A**	Non-Potable Water	
Inorganic WetChem			
Alkalinity	SM2320B**	Non-Potable Water	
CBOD	SM 5210B	Non-Potable Water	
COD	SM5220C	Non-Potable Water	
BOD	SM5210B	Non-Potable Water	
Color, Apparent	SM2120B	Non-Potable Water	
Ion Chromatography (Bromide,	EPA 300.0**, SW846 9056A**	Non-Potable Water	
Fluoride, Chloride, Sulfate, Nitrite,	,, ,		
Nitrate,) – Aqueous			
Nitrate/Nitrite	EPA 353.2**	Non-Potable Water	
Total Kjeldahl Nitrogen	EPA 351.2**	Non-Potable Water	
Ammonia	EPA 350.1**	Non-Potable Water	
Oil & Grease, Gravimetric – AQ	EPA 1664A**, SW846 9070A**	Non-Potable Water	
Orthophosphate	EPA 365.3**	Non-Potable Water	
Nitrate	SM 4500NO2-B	Non-Potable Water	
pH by electrode (Waters)	SM4500H+B**; SW846 9040C**	Non-Potable Water	
Specific Conductance	EPA 120.1	Non-Potable Water	
Nitrate-Nitrite	SM 4500 NO3-E	Non-Potable Water	
Sulfide	SM4500S=F**	Non-Potable Water	
Chloride	SM 4500 CI-B	Non-Potable Water	
Total Dissolved Solids	SM2540C**	Non-Potable Water	
Total Organic Carbon	SM5310B**, SW846 9060A**	Non-Potable Water	
Total Phosphorus	EPA 365.3	Non-Potable Water	
Total Solids	SM2540B**	Non-Potable Water	
Total Suspended Solids	SM2540D**	Non-Potable Water	
Turbidity	EPA 180.1	Non-Potable Water	
Total CN	EPA 335.4, SW846 9012B**	Non-Potable Water	
Un-Ionized Ammonia - calculation	FDE SOP10/03/83	Non-Potable Water	
Perchlorate	EPA 314	Non-Potable Water	
Calcium Hardness by Calculation	SM18 2340B	Non-Potable Water	
Hardness, Total by Calculation	SM18 2340B	Non-Potable Water	
MBAS (Anionic Surfactants as)	SM5540C	Non-Potable Water	



Method Type	Method Number	Regulatory Program
Corrosivity & pH – aqueous Hexavalent Chromium	SW846 9040C** SW846 7196A**	Non-Potable Water Non-Potable Water
Organics		
EDB and DBCP	SW846 8011 Mod**	Solid and Chemical Material
Volatile Organics	SW846 8260B**	Solid and Chemical Material
Semi-Volatile Organics	SW846 8270D**	Solid and Chemical Material
Semi-Volatile Organics	SW846 8270D SIM**	Solid and Chemical Material
Gasoline Range Organics	SW846 8015C,D**	Solid and Chemical Material
Diesel Range Organics	SW846 8015C,D**	Solid and Chemical Material
Alcohols	SW846 8015C,D**	Solid and Chemical Material
Polynuclear-Aromatic Hydrocarbons	SW846 8310**	Solid and Chemical Material
Explosives	SW846 8330A**, 8332**	Solid and Chemical Material
Explosives	SW846 8330B**	Solid and Chemical Material
Organochlorine Pesticides	SW846 8081B**	Solid and Chemical Material
Polychlorinated Biphenyls	SW846 8082A**	Solid and Chemical Material
Chlorinated Herbicides	SW846 8151A**	Solid and Chemical Material
Organophosphorus Pesticides	SW846 8141B**	Solid and Chemical Material
Perchlorate	SW-846 6850	Solid and Chemical Material
Total Petroleum Hydrocarbons	FLPRO**	Solid and Chemical Material
Tennessee EPH	TN-EPH**	Solid and Chemical Material
Tennessee GRO	TN-GRO**	Solid and Chemical Material
Wisconsin DRO	WI-DRO**	Solid and Chemical Material
Petroleum Hydrocarbons	Iowa OA-1**	Solid and Chemical



Method Type	Method Number	Regulatory Program
Petroleum Hydrocarbons	Iowa OA-2**	Material Solid and Chemical Material
Volatile Petro. Hydrocarbons	Massachusetts VPH, 2004**	Solid and Chemical Material
Extractable Petro. Hydrocarbons	Massachusetts EPH, 1998**	Solid and Chemical Material
Total Petroleum Hydrocarbons	TX-1005**	Solid and Chemical Material
Acrylamide	SW846 8316	Solid and Chemical Material
Metals		
ICP: General – EPA WW	SW846 6010C**	Solid and Chemical
Cold Vapor Mercury – EPA DW	SW846 7471B**	Material Solid and Chemical Material
Inorganic WetChem		
Ion Chromatography (Bromide, Fluoride, Chloride, Sulfate, Nitrite, Nitrate,) – Aqueous	SW846 9056A**	Solid and Chemical Material
Oil & Grease, Gravimetric – Solid	SW846 9071A**	Solid and Chemical Material
Total CN	SW846 9012B**	Solid and Chemical Material
Total Organic Carbon	SW846 9060A**	Solid and Chemical Material
Ammonia	EPA 350.1	Solid and Chemical
Total Kjeldahl Nitrogen	EPA 351.2	Material Solid and Chemical
Total Phosphorus	EPA 365.3	Material Solid and Chemical
Waste Ignitability	SW846 1010A**	Material Solid and Chemical
Hexavalent Chromium/soils	SW846 7196A**	Material Solid and Chemical
Corrosivity & pH – aqueous	SW846 9040C**	Material Solid and Chemical
Corrosivity & pH – solid	SW846 9045D**	Material Solid and Chemical Material



Method Type	Method Number	Regulatory Program
Cyanide Reactivity	SW846 Chapter 7**	Solid and Chemical Material
Sulfide Reactivity	SW846 Chapter 7**	Solid and Chemical Material
Organics		
Volatile Organics	TO-3	Air and Emissions
Preparation Methods*		
Liquid/Liquid Extraction, Water	SW846 3510C	
Solid Phase Extraction, Water	SW846 3535A	
Solids Extraction by Sonication	SW846 3550B	
Microwave-assisted extraction, solids	SW846 3546	
Acid/Base Partitioning	SW846 3650B	
Sulfur Cleanup of Extracts	SW846 3660B	
Sulfuric Acid Cleanup	SW846 3665A	
Purge & Trap - Aqueous	SW846 5030B	
Purge & Trap – Solids	SW846 5035A	
Total Recoverable Metals Digestion	EPA 200.7	
Non-Pot. Water Digest: ICP	SW846 3010A	
Alkaline Digestion of Soils for	SW846 3060A	
Hexavalent Chromium	011101000000	
Digestion of Soils for ICP	SW846 3050B	
TCLP	SW846 1311	
SPLP	SW846 1312	

^{*} Preparation methods are not listed on Primary TNI Accreditation per State of Florida DOH rules. However, for the benefit of other accrediting authorities, these methods are inspected during FDOH visits. Listing of surveyed and approved preparation methods is available from on-site inspection report.

^{**} Methods certified by DoD ELAP



Non-TNI-Accredited Fields of Testing

Method Type	Method Number	Regulatory Program	
Organics			
Thiodiglycol	Accutest in-house method (HPLC)	Solid and Chemical Material	
N-Nitroso-N-Ethylurea	Accutest in-house method (HPLC)		
Volatile Petroleum Hydrocarbons	Missouri Gasoline Range Organics	Solid and Chemical Material	
Extractable Hydrocarbons	Missouri Diesel Range Organics	Solid and Chemical Material	
Extractable Hydrocarbons	Missouri Oil Range Organic	Solid and Chemical Material	
Volatile Petroleum Hydrocarbons	Alaska AK-101**	Solid and Chemical Material	
Extractable Hydrocarbons	Alaska AK-102**	Solid and Chemical Material	
Extractable Hydrocarbons	Alaska AK-103**	Solid and Chemical Material	
Volatile Petroleum Hydrocarbons	OK GRO**	Solid and Chemical Material	
Extractable Hydrocarbons	OK DRO**	Solid and Chemical Material	
Inorganic WetChem			
Oxidation-Reduction Potential	ASTM D1498-76, mod. for solids	Solid and Chemical Material	
Percent Ash (dry basis)	ASTM D2974-87, D482-91	Solid and Chemical Material	
Grain Size (hydrometer)	ASTM D422-63	Solid and Chemical Material	
Sieve Testing	ASTM D422-63	Solid and Chemical Material	
Specific Gravity	ASTM D1298-85	Solid and Chemical Material	
Acidity Dissolved Oxygen Mineral Suspended Solids Organophosphonic Acids	SM2310B EPA 360.1 EPA 160.2/160.4 Accutest in-house method (IC)	Non-Potable Water Non-Potable Water Non-Potable Water Solid and Chemical Material	



Method Type	Method Number	Regulatory Program
Perchlorate	EPA 314MOD	Solid and Chemical Material
Percent Solids	SM19 2540G	Solid and Chemical Material
Settleable Solids	EPA 160.5	Non-Potable Water
Total Mineral Solids	EPA 160.4	Non-Potable Water
Total Residual Chlorine	EPA 330.5	Non-Potable Water
Total Volatile Solids	EPA 160.4	Non-Potable Water
Volatile Suspended Solids	EPA 160.2/160.4	Non-Potable Water
CÑ Amenable to Chlorination	EPA 335.4	Solid and Chemical Material
Bicarbonate, Carbonate, CO2 - calculation	SM19 4500 CO2D	Non-Potable Water
Ferrous Iron	SM19 3500 FE-D	Non-Potable Water
Salinity - calculation	SM19 2520B	Non-Potable Water
Paint Filter Test	SW846 9095	Solid and Chemical Material
Corrosivity towards steel	SW846 1110	Solid and Chemical Material
Corrosivity & pH – aqueous	SW846 9040C	Solid and Chemical Material



Appendix III

Equipment List



ORGANIC INSTRUMENTATION

Instrument	Model	Location	Serial #	Year
GC/MS	Agilent 5975C MSD/OI 4551/4660	MS-VOA	US11172705	2011
GC/MS	Agilent 5975C MSD/OI 4551/4660	MS-VOA	US11322911	2011
GC/MS	Agilent 5975C MSD/OI 4551/4660	MS-VOA	US10102029	2010
GC/MS	Agilent 5975C MSD/OI 4551/4660	MS-VOA	US83120965	2008
GC/MS	Agilent 5975N MSD/Agilent 7683 AS	SVOC Lab	US71225975	2007
GC/MS	Agilent 5975N MSD/Agilent 7683 AS	SVOC Lab	US62724401	2006
GC/MS	Agilent 5975N MSD/Agilent 7683 AS	SVOC Lab	US53921303	2005
GC/MS	Agilent 5973N MSD/Agilent 7683 AS	SVOC Lab	US40620599	2004
GC/MS	Agilent 5973 MSD/OI 4660/4552 Archon	MS-VOA	US41746628	2004
GC/MS	Agilent 5973 MSD/OI 4660/4552 Archon	MS-VOA	US41746633	2004
GC/MS	Agilent 5973 MSD/OI 4560/4552 Archon	Soil VOA	US21843765	2002
GC/MS	Agilent 5973 MSD/OI 4551/4660	MS-VOA	US21844034	2002
GC/MS	Agilent 5973 MSD/OI 4660/4552 Archon	MS-VOA	US02440350	2000
GC/MS	Agilent 5973 MSD/OI 4560/4552 Archon	MS-VOA	US94240108	1999
GC/MS	Agilent 5973 MSD/Agilent 7683 AS	SVOC Lab	US82311290	1998
GC/MS	Agilent 5973 MSD/Agilent 7683 AS	SVOC Lab	US81211109	1998
GC/MS	Hewlett-Packard 5970 MSD/OI 4560/4552 Archon	Soil VOA	3034A12782	1989
GC/MS	Hewlett-Packard 5970 MSD/OI 4560/4552 Archon	Soil VOA	2905A11904	1987
GC/MS	Hewlett-Packard 5970 MSD/OI 4560/4552 Archon	Soil VOA	2716A10454	1987
GC	Agilent 7890A/Dual ECD/7683B AS	SVOC Lab	CN10842133	2008
GC	Agilent 7890A/Dual FID/7683B AS	SVOC Lab	CN10902149	2009
GC	Agilent 7890A/Dual FID/7683B AS	SVOC Lab	CN10716029	2009
GC	Agilent 7890A/Dual ECD/7683B AS	SVOC Lab	CN10741128	2007



Instrument	Model	Location	Serial #	Year
GC	Agilent 6890/Dual FPD/7683B AS	SVOC Lab	US10643024	2006
GC	Agilent 6890/Dual FID/7683B AS	SVOC Lab	CN10641049	2006
GC	Agilent 6890/Dual ECD/7683B AS	SVOC Lab	CN10641081	2006
GC	Agilent 6890/Dual ECD/7683B AS	SVOC Lab	US10613003	2006
GC	Agilent 6890/PID/PID/OI 4560/4552 Archon	GC VOA	CN10421047	2004
GC	Agilent 6890/PID/FID/ENTECH 7032A-LB	GC VOA	US10239007	2002
GC	Agilent 6890N/Dual FID/HP 7683 AS	SVOC Lab	CN10425061	2004
GC	Agilent 6890N/Dual ECD/HP 7683 AS	SVOC Lab	US10333015	2003
GC	Agilent 6890/Dual ECD/HP 7683 AS	SVOC Lab	US00036916	2000
GC	Agilent 6890/Dual ECD/HP 7683 AS	SVOC Lab	US00028304	1999
GC	Hewlett-Packard 5890/PID/FID/ OI 4560/4552 Archon	GC VOA	3336A60617	1993
GC	Hewlett-Packard 5890/Dual FID/HP 7673 AS	SVOC Lab	3336A59489	1993
GC	Hewlett-Packard 5890/PID/FID/ OI 4560/4552 Archon	GC VOA	3336A51045	1993
GC	Hewlett-Packard 5890/PID/FID/OI 4560/4552 Archon	GC VOA	3203A41646	1992
GC	Hewlett-Packard 5890/PID/FID/OI 4560/4552 Archon (screening instrument)	GC VOA	3223A4267	1992
GC	Hewlett-Packard 5890/Dual FID/HP 7673 AS	SVOC Lab	3126A51085	1991
GC	Hewlett-Packard 5890/PID/FID/ dual MPM 16	Soil VOA	3029A29748	1990
GC	Hewlett-Packard 5890/FID	Soil VOA	2843A20183	1988
GC	Hewlett-Packard 5890/Dual FID	GC VOA	2728A12705	1987
HPLC	Agilent 1100 Automated LC System	HPLC Room	DE91606857	1999
HPLC	Agilent 1100 Automated LC System	HPLC Room	DE23917648	2002
HPLC	Agilent 1100 Automated LC System	HPLC Room	DE01608404	2000
HPLC	Agilent 1100 Automated LC System	HPLC Room	DE40522115	2004
HPLC	Agilent 1100 Automated LC System	HPLC Room	DE03000863	2003



Instrument	Model	Location	Serial #	Year
HPLC	Agilent 1100 Automated LC System	HPLC Room	DE61800775	2006
O-Prep	ESSA LM2-P Ring and Puck mill	Explosives Prep Lab	215090-004	2008
O-prep	Microwave extractor	Organic Prep Lab	MD3482	2010
O-Prep	TurboVap 4 units	Organic Prep Lab		2001
O-Prep	TurboVap 3 units	Organic Prep Lab		2004
O-Prep	TurboVap 1 unit	Organic Prep Lab		2007
O-Prep	Sonicator 2 units	Organic Prep Lab		2004
O-Prep	Sonicator 3 units	Organic Prep Lab		2007
O-Prep	Midi-Vap 2000 Kontes	Organic Prep Lab	479200-2000	2000
Data	Hewlett-Packard/MS ChemStation	Labwide		1999, with
System				subsequent
				upgrades

Inorganic Instrumentation

Instrument	Model	Location	Serial #	Year
ICP	Thermo ICAP 6000 Series	Metals Lab	20100903	2010
ICP	Thermo ICAP 6000 Series	Metals Lab	20103825	2010
Mercury Analyzer	Leeman Hydra AA	Metals Lab	HA-2022	2002
Mercury Analyzer	Leeman Hydra AA II	Metals Lab	2004	2012
TOC Analyzer	Shimadzu	WetChem IC room	H51404235007	2004
TOC Analyzer	Shimadzu	WetChem IC room	H51404735099	2010
IC	Dionex IC-2100	WetChem IC room	10110002	2010
IC	Dionex IC-2000	WetChem IC room	04070250	2004
Auto Analyzer	QuickChem 8500 Series	WetChem main room	050500000130	2005
Auto Analyzer	QuickChem 8500 Series 2	WetChem main room	111200001380	2011
Spectrophotometer	Milton-Roy Spectronic 200	WetChem main room	2 units	2000
Digestion block	DigiPrep	WetChem main room	4 units	2005



Centrifuge	CentraCL2	WetChem main room	42613052	2003
MicroDistillation Block	Lachat	WetChem main room	2 units	2005

LIMS		
Instrument	Model	Year
LIMS	HP True 64	1999



Appendix IV

Certification Summary



Certifying Authority	Certification Program	Registration No.
Alaska	Contaminated Sites	UST-088
Arkansas	Solid/Hazardous Wastes, Non-Potable Water	88-0620
California (NELAP)	Potable Water, Solid/Hazardous Waste	04226CA
Department of	Non-Potable Water, Solid and Chemical Materials	L-2229
Defense (DoD)		
Florida (NELAP)	Potable, Non-Potable, Solid Waste, UST, Air Toxics	E83510
Georgia	Solid/Hazardous Wastes	Not Applicable
Illinois	Solid/Hazardous Wastes, Non-Potable Water	
Iowa	UST, Solid/Hazardous Wastes, Non-Potable Water	IA366
Kansas (NELAP)	Solid/Hazardous Wastes, Non-Potable Water	E-10327
Kentucky	Underground Storage Tank Program	0065
Louisiana (NELAP)	Solid/Hazardous Wastes	38582
Massachusetts	Non-Potable Water	M-FL946
Mississippi	Potable Water	Not Applicable
Nevada	Non-Potable Water, Solid/Hazardous Wastes	FL009462008A
New Jersey (NELAP)	Solid/Hazardous Wastes, Non-Potable Water	FL002
North Carolina	Solid/Hazardous Wastes, Non-Potable Water	573
Oklahoma	Non-Potable Water, Solid/Hazardous Waste	9959
South Carolina	Solid/Hazardous Wastes, Non-Potable Water	96038001
Texas (NELAP)	Non-Potable Water, Solid/Hazardous Waste	T104704040-08-
		TX
US Dept. of	Foreign Soils Permit	S-56027
Agriculture		
Utah (NELAP)	Potable, Non-Potable, Solid/Chemical Materials	FL009462008A
Virginia (NELAP)	Potable, Non-Potable, Solid/Chemical Materials	460177
Washington	Potable, Non-Potable, Solid/Chemical Materials, Air	C2046
Wisconsin	Solid/Hazardous Wastes, Non-Potable Water	399043370



Appendix V

SOP List



OP029

OP030

Samples

SOP # TITLE

Organic Preparation Department

OP002 SOP for Glassware Cleaning and Storage **OP003** SOP for Reagent Prep SOP for the Extraction of Semi-volatile Organics (BNAs) from Aqueous **OP006** Samples **OP007** SOP for the Extraction of Semi-volatile Organics (BNAs) from Solid Samples **OP008** SOP for the Extraction of Pesticides/PCBs from Aqueous Samples **OP009** SOP for the Extraction of Pesticides/PCBs from Solid Samples SOP for the Extraction of Pesticides/PCBs from Solid Samples, microwave OP009MW SOP for the Extraction of Diesel Range Organics (DRO) from Aqueous **OP010** Samples **OP011** SOP for the Extraction of Diesel Range Organics (DRO) from Solid Samples **OP011MW** SOP for the Extraction of Diesel Range Organics (DRO) from Solid Samples SOP for the Extraction of Petroleum Related Organics (FL-PRO) from **OP012** Aqueous Samples **OP013** SOP for the Extraction of Petroleum Related Organics (FL-PRO) from Solid Samples **OP014** SOP for the Extraction of PAHs from Aqueous Samples (HPLC) SOP for the Extraction of PAHs from Solid Samples (HPLC) **OP015 OP016** SOP for the Extraction of EDB/DBCP from Aqueous Samples **OP017** SOP for the Extraction of EDB/DBCP from Solid Samples **OP018** SOP for the Extraction of Explosives from Aqueous Samples **OP019** SOP for the Extraction of Explosives from Solid Samples **OP020** SOP for Sample Introduction via SW846-5035 **OP021** SOP for Sample Introduction via SW846-5030B **OP022** SOP For The Extraction Of Nitroglycerine And Pentaerythritoltetranitrate (PETN) From Water Samples (HPLC Analysis) SOP For The Extraction Of Nitroglycerine And Pentaerythritoltetranitrate **OP023** (PETN) From Solid Samples (HPLC Analysis) Standard Operating Procedure For The Extraction Of Nitroaromatics From **OP024** Water Samples **OP025** SOP For Sample Preparation For Dissolved Gases In Aqueous Samples **OP026** SOP For The Extraction Of Extractable Petroleum Products (OA-2) From Water Samples SOP For The Extraction Of Extractable Petroleum Products (OA-2) From **OP027** Solid Samples **OP028** SOP For The Extraction Of Diesel And Oil Range Organics From Water Samples

SOP For The Extraction Of Diesel And Oil Range Organics From Solid

SOP For The Extraction Of Extractable Petroleum Hydrocarbons From



Water Samples (Tennessee EPH)

OP031 SOP For The Extraction Of Extractable Petroleum Hydrocarbons From Solid

Samples (Tennessee EPH)

OP032 SOP For The Extraction Of Volatile Petroleum Hydrocarbons From Soil

Samples, MA-VPH

OP033 SOP For The Extraction Of PCBs From Wipes

OP034 SOP For The Extraction Of Diesel Range Organics (DRO) From Aqueous

Samples WI-DRO

OP035 SOP For The Extraction Of Massachusetts Extractable Petroleum

Hydrocarbons From Water Samples

OP036 SOP For The Extraction Of Massachusetts Extractable Petroleum

Hydrocarbons From Solid Samples

OP037 SOP For The Extraction Of Chlorinated Herbicides From Water Samples SOP For The Extraction Of Chlorinated Herbicides From Soil Samples SOP For The Extraction Of Chlorinated Herbicides From Soil Samples,

microwave

OP039 SOP For The Solid Phase Extraction (SPE) Cartridge Cleanup Of Pesticide

Extracts

OP040 SOP For SPLP Leaching Of SVOC And Metals

OP041 SOP For TCLP Leaching Of VOC

OP042 SOP For SPLP Leaching Of SVOC And Metals

OP043 SOP For SPLP Leaching Of VOC

OP044 SOP For The Extraction Of Organophosphorus Pesticides From Water

Samples

OP044SP SOP For The Extraction Of Organophosphorus Pesticides From Water

Samples, Solid Phase Extraction

OP045 SOP For The Extraction Of Organophosphorus Pesticides From Soil

Samples

OP045MW SOP For The Extraction Of Organophosphorus Pesticides From Soil

Samples, microwave

OP046 SOP for the Extraction of Explosives from Solid Samples, SW-8330B SOP for the Extraction of Explosives from Aqueous Samples, SW-8330B

OP048 SOP for the Extraction of PCB Congeners from Aqueous Samples SOP for the Extraction of PCB Congeners from Solid Samples

OP050 SOP For The Extraction Of Alaska Extractable Petroleum Hydrocarbons

From Water Samples

OP051 SOP For The Extraction Of Alaska Extractable Petroleum Hydrocarbons

From Solid Samples

OP052 SOP For The Extraction Of Oklahoma Extractable Petroleum Hydrocarbons

From Water Samples

OP053 SOP For The Extraction Of Oklahoma Extractable Petroleum Hydrocarbons

From Solid Samples

OP054 SOP For The Extraction Of 1,4-Dioxane From Water Samples

OP055 SOP For The Extraction Of Petroleum Hydrocarbons From Water Samples,



COD #	TITLE
SOP#	TITLE

TX-1005

OP056 SOP For The Extraction Of Petroleum Hydrocarbons From Solid Samples,

TX-1005

OP057 SOP for Sample Introduction via AK-101

Gas Chromatography/ HPLC SOPs

GC002	Analysis Of 1,2-Dibromoethane (EDB) And 1,2-Dibromo-3-Chloropropane (DBCP) By Gas Chromatography, Electron Capture Detector
GC004	Aromatic Volatiles By Gas Chromatography Using PID Detectors EPA 602
GC005	Analysis Of Organochlorine Pesticides By Gas Chromatography, Electron
	Capture Detector EPA 608
GC006	Analysis Of Polychlorinated Biphenyls By Gas Chromatography, Electron
00000	Capture Detector EPA 608
GC007	Analysis Of Polynuclear Aromatic Hydrocarbons By Gas Chromatography,
00001	Flame Ionization Detector EPA 610
GC008	Analysis Of Petroleum Range Organics By Gas Chromatography Using
00000	Flame Ionization Detector
GC009	Analysis Of 1,2-Dibromoethane (EDB) And 1,2-Dibromo-3-Chloropropane
00003	(DBCP) By Gas Chromatography, Electron Capture Detector SW-846 8011
GC010	Analysis Of Gasoline Range Organics By Gas Chromatography Using Flame
GCUTU	Ionization Detector
GC011	Analysis Of Diesel Range Organics By Gas Chromatography Using Flame
GCUTT	Ionization Detector
GC014	Analysis Of Polychlorinated Biphenyls By Gas Chromatography, Electron
00014	Capture Detector SW-846 8082
GC015	Analysis Of Organochlorine Pesticides By Gas Chromatography, Electron
00013	Capture Detector SW-846 8081
GC016	Analysis Of Nitroaromatics And Nitramines By HPLC
GC017	Aromatic Volatiles By Gas Chromatography Using PID Detectors SW-8021
GC018	Analysis Of Polynuclear Aromatic Hydrocarbons By HPLC SW-846 8310
GC019	Analysis Of Dissolved Gases By Gas Chromatography, Flame Ionization
00000	Detector
GC020	Analysis Of Nitroglycerine And PETN By HPLC
GC021	Analysis Of Volatile Petroleum Hydrocarbons By Gas Chromatography
GC022	Analysis Of Extractable Petroleum Products By Gas Chromatography Using
	Flame Ionization Detector OA-2
GC023	Analysis Of Diesel And Oil Range Organics By Gas Chromatography Using
	Flame Ionization Detector
GC024	Analysis Of Petroleum Hydrocarbons By Gas Chromatography Using Flame
	Ionization Detector (Tennessee EPH)
GC025	Analysis Of Nitroaromatics By Gas Chromatography Using Electron Capture
	Detector
GC026	Method For Determination Of Volatile Petroleum Hydrocarbons By GC-



QA003

TITLE SOP# PID/FID GC027 Analysis Of Non-Halogenated Organics By Gas Chromatography Using Flame Ionization Detector GC028 Analysis Of Gasoline Range Organics By Gas Chromatography Using Flame Ionization Detector TDEC GRO GC029 Analysis Of Diesel Range Organics By Gas Chromatography Using Flame Ionization Detector Wi DRO Analysis Of Extractable Petroleum Hydrocarbons By Gas Chromatography GC030 Using Flame Ionization Detector MA-EPH GC031 Analysis Of Chlorinated Herbicides Using GC-ECD GC032 Analysis Of Organophosphorus Pesticides Using GC-NPD Or FPD GC033 Air Analysis By GC-PID/FID GC034 Analysis Of Nitroaromatics, Nitramines And Nitrate Esters By HPLC Method 8330b GC035 Screening Of Volatile Organics By GC-PID/FID GC036 Analysis of PCB Congeners by ECD GC037 Analysis of Diesel and Oil Range Organics by GC/FID, AK-102, AK-103 GC038 Analysis of Gasoline Range Organics by GC/FID, AK-101 GC039 Analysis of Diesel Range Organics by GC/FID, OK-GRO GC040 Analysis of Gasoline Range Organics by GC/FID, OK-GRO GC041 Analysis of N-Nitroso-N-Ethylurea by HPLC GC042 Analysis of Thiodiglycol by HPLC GC043 Analysis of Acrylamide by HPLC GC044 Analysis of Petroleum Organics by TX-1005 **Mass-Spectrometry SOPs** MS003 Analysis of Volatile Organics by EPA Method 624 MS004 Analysis of Semi-volatile Organics by EPA Method 625 Analysis of Volatile Organics by EPA Method 8260B MS005 MS006 Analysis of Semi-volatile Organics by EPA Method 8270C MS008 Analysis of Semi-volatile Organics by EPA Method 8270C SIM MS009 Analysis of Volatile Organics by GC/MS MS010 Analysis of Volatile Organics by GC/MS SIM MS011 Analysis of Semi-volatile Organics by EPA Method 8270D MS012 Analysis of 1,4-Dioxane by EPA 522 MS013 Analysis of Perchlorate by SW-846 6850 **Quality Assurance SOPs QA001** Preparation, Approval, Distribution & Archiving Of Standard Operating Procedures (SOPs) Calibration Of Thermometers QA002

Personnel Training And Analyst Proficiency



QA004 Temperature Monitoring

QA005 Calibration Of Analytical Balances
QA006 Eppendorf Pipette Calibration
QA007 Sample Batching Procedure
QA008 Creating New Accounts
QA009 Creating New Projects

QA010 Confidentiality Protection Procedures

QA011 Signature Authority

QA012 Employee Technical Ethics Responsibilities
QA013 Client Complaint Resolution Procedure

QA014 Procedures For The Purchase Of Laboratory Supplies

QA015 Procedures For The Preparation, Distribution, Use And Archiving Of

Laboratory Logbooks

QA016 Corrective Action Procedure

QA017 Standards Traceability Documentation Procedure

QA018 Procedure For Login, Management, Handling, And Reporting Of Proficiency

Test (Pt) Samples

QA019 Quality System Review

QA020 Procedure For Developing Method Performance Criteria And Experimental

Method Detection Limits

QA021 Subcontracting Procedures
QA022 Internal Audit Procedure
QA023 Fume Hood Inspection
QA027 Review Of Inorganics Data
QA028 Review Of Organics Data

QA029 Manual Integration Of Chromatographic Peaks

QA030 Procedure For The Development And Use Of in-house Quality Control

Criteria

QA031 Air Quality Monitoring Of Extraction Laboratory

QA032 Routine Maintenance For Major Analytical Instrumentation

QA033 Laboratory Safety
QA034 Sample Homogenizing
CA035 Salvent Testing And Ang

QA035 Solvent Testing And Approval QA036 Data Package Generation

QA037 Deionized Water Quality Control Procedure

QA038 Data Integrity Training Procedure
QA039 Data Integrity Monitoring Procedure

QA040 Procedure For Conducting Data Integrity Investigations

QA041 Procedure For The Confidential Reporting Of Data Integrity Issues

QA042 Basic Calculations For General Chemistry Methods

QA043 Data Qualifier SOP

QA044 Calibration Of Micro-Distillation Tubes

QA045 Estimation of Uncertainty

QA046 Document Control



QA047 Management of Client Project

QA048 Data Entry for Log-In

General Chemistry SOPs

GNSOP: 101 Acidity (pH 8.2)

GNSOP: 102 Alkalinity, Total (pH 4.5)

GNSOP: 103 Ammonia – Distillation Procedure

GNSOP: 104 Nitrogen, Ammonia

GNSOP: 105 Bicarbonate, Carbonate, Free Carbon Dioxide

GNSOP: 106 Chemical Oxygen Demand

GNSOP: 107 Chloride by Titration

GNSOP: 109 Color, Apparent

GNSOP: 110 Chromium, Hexavalent (Water)

GNSOP: 113 Cyanide Distillation/Aqueous And Solid Samples

GNSOP: 115 Cyanide, Total **GNSOP: 116** Dissolved Oxygen

GNSOP: 121 Ignitability

GNSOP: 122 Anionic Surfactants As MBAS

GNSOP: 123 Nitrogen, Nitrite **GNSOP: 126** Ortho Phosphate

GNSOP: 127 Paint Filter Liquids Test

GNSOP: 128 Phenols Distillation, Soil And Water Samples

GNSOP: 130 Phenols, Total Recoverable

GNSOP: 133 Settleable Solids

GNSOP: 134 Total Suspended Solids (Non Filterable Residue)GNSOP: 135 Total Dissolved Solids (Total Filterable Residue)

GNSOP: 136 Reactive Sulfide And Reactive Cyanide

GNSOP: 137 pH By Electrode - Water

GNSOP: 140 Sulfide

GNSOP: 144 Total Phosphorus

GNSOP: 145 Turbidity

GNSOP: 147 Winkler Titration For DO Standardization

GNSOP: 161 Percent Solids

GNSOP: 163 Specific Conductance At 25 C.

GNSOP: 166 pH By Electrode – Soil

GNSOP: 167 Biochemical Oxygen Demand (BOD)

GNSOP: 171 Hexachromium In Soils

GNSOP: 179 Corrosivity (Soil pH By Electrode)

GNSOP: 182 Total Kjeldahl Nitrogen **GNSOP: 189** Corrosivity Toward Steel

GNSOP: 190 Total Nitrogen, Organic Nitrogen

GNSOP: 191 Nitrogen, Nitrate

GNSOP: 192 Carbonaceous Biochemical Oxygen Demand (CBOD)



GNSOP: 193	3	Oxidation-Reduction Potential
ONICOD 40	4	

GNSOP: 194 Ferrous Iron

GNSOP: 196 Glassware Cleaning

GNSOP: 197 Anions By Ion ChromatographyGNSOP: 211 Oil & Grease And PHC By 1664GNSOP: 212 Fractional Organic Carbon

GNSOP: 213 Walkley-Black Total Organic Carbon

GNSOP: 214 Particle Size By Sieve

GNSOP: 215 TOC In Water

GNSOP: 216 Particle Size By Hydrometer

GNSOP: 218 Perchlorate **GNSOP: 219** Bulk Density

GNSOP: 222 Un-Ionized Ammonia Calculation

GNSOP: 224 Hardness By Calculation

GNSOP: 225 Cation Exchange Capacity Of Soils (Sodium Acetate)

GNSOP: 226 TOC In Soil

GNSOP: 227 Oil And Grease – Gravimetric Analysis (Soils)
 GNSOP: 228 Anions By Ion Chromatography - IC 2000
 GNSOP: 229 Determination Of Nitrocellulose In Water
 Determination Of Nitrocellulose In Soil

GNSOP: 231 % Ash

SAM101

GNSOP: 232 Determination Of Nitrate and Nitrite by Lachat

Sample Receipt And Storage

Metals SOPs

MET 100	Metals By Inductively Coupled Plasma
MET 103	Digestion Of Water Samples For Flame And ICP Analysis
MET 104	Digestion Of Soils For ICP Analysis
MET 105	Cold Vapor Analysis Of Mercury For Soils
MET 106	Cold Vapor Analysis Of Mercury For Water Samples

Sample Management SOPs

CANTO	Sample Necelpt And Storage
SAM102	Procedure For Sample Bottle Preparation And Shipment
SAM104	Sample Container Quality Control
SAM108	Sample And Laboratory Waste Disposition
SAM109	Foreign Soil receipt and Handling

7	THE FORMI	ER ST. LO	UIS ORDN	JANCE PLA	NT
SITE-SPE	CIFIC SITE	SAFETY A	AND HEAL	TH PLAN	ADDENDUM

ST. LOUIS ORDNANCE PLANT SITE-SPECIFIC SSHP ADDENDUM

This addendum to the Regional Site Safety and Health Plan (SSHP) for Regional LTO/LTM for Seven Installations provides specific guidelines for field activities for the former St. Louis Ordnance Plant (SLOP). This addendum must be used in conjunction with the Regional APP/SSHP. As such, the APP and SSHP must be read, understood, and the Plan Acknowledgement Sheets signed prior to field activities.

Emergency Information: In the event of any situation or unplanned occurrence requiring assistance, follow the HGL Incident Reporting Procedures (APP Appendix F), outlined below:

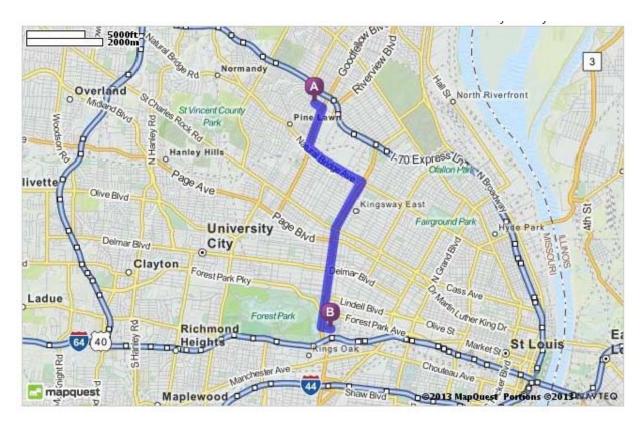
- For critical or life threatening injury, please call 911.
- Contact WorkCare as soon as care for injury and time permits: (888) 449-7787.
- Remember to keep your Supervisor and Project Manager informed.
- Accident and Injury Actions
 - o Prevent further harm. Move victim to a safe place if injury will not result by moving. Do not endanger your safety.
 - o Provide First Aid as required.
 - o Call for outside assistance if needed (911, base incident command, or WorkCare).
 - Communicate with Project Manager, Office Manager, Corporate Health and Safety: (800) 341-3674.

Emergency Telephone Numbers and Project Contacts

Fire, Police, Emergency Medical Services	es 911		
Emergency Medical Care	Barnes Jewish Hospital		
	1 Barnes Jewish	(214) 747 2000	
	Hospital Plaza.	(314) 747-3000	
	St. Louis, MO		
National Poison Control	(800) 222	2-1222	
National Response Center Environmental Emergencies	(800) 42-	4-8802	
EPA Spill & Release Notification	(800) 424	4-9346	
Client			
USACE Project Manager	Josephine Newton-Lund	(816) 389-3912	
AEC	Barry McFarland	(316) 681-1759	
Utility Locater Services	Missouri One-Call 811		
HydroGeo	Logic, Inc. Contacts		
H&S Emergency Number	(800) 341-3647		
Project Manager	Chris Williams	Office: (913) 647-2536	
	CIII IS WIII allis	Cell: (816) 204-1861	
Field Supervisor (FS)/Site Safety and	Klaas Doeden	Office: (913) 378-2301	
Health Officer (SSHO)	Kiaas Docucii	Cell: (816) 547-5013	
Safety and Health Manager (SHM)	Mary Ann Heaney, CIH	Office: (303) 665-8528	
	Waiy Aim Healicy, CIII	Cell: (303) 250-7753	
HGL Corporate Occupational Physician	Peter Greaney, MD	(800) 455-6155	
WorkCare	Work Care 27/7 Hotline (888) 449-7787		

Directions to nearest hospital: From 6400 Stratford Ave., proceed southeast on Stratford for approximately 1.0 mile. Turn right onto Goodfellow Blvd. Proceed approximately 0.8 mile and turn left onto Natural Bridge Ave. Continue 1.2 miles on Natural Bridge Ave. then turn right onto N. Kingshighway Blvd. Proceed approximately 3.0 miles to Barnes-Jewish Hospital Plaza and turn left. Barnes Jewish Hospital, located at 1 Barnes Jewish Hospital Plaza, St. Louis, MO, is on the left.

Drive time: 14 minutes



<u>Contingency Plans for Severe Weather:</u> Procedures are outlined in Section 9.u of the APP. The main rally point will be determined daily during the morning TSM depending on the location of the day's activities.

<u>Site Description</u>: LTM activities at the former SLOP will be conducted at OU1. Table SLOP-1 shows the anticipated field activities and the applicable AHAs.

<u>General Hazards:</u> The potential general physical, chemical, and biological hazards are discussed in Section 3 of the Regional SSHP. Radiological hazards and MEC hazards are not anticipated.

<u>Site-Specific Chemical Hazards</u>: Exposure to chemical hazards may occur from the site contaminants of concern and materials brought on site as part of the work effort. The risk to personnel will vary by activity. It is anticipated that contaminants will not reach levels considered to be hazardous during site activities. **Contaminants that may be encountered,**

and their properties and acute health effects are provided in Table SLOP-2. The highest observed contaminant concentrations shown on the table were taken from the July 2012 Groundwater Monitoring Report.

Additional substances that may be brought to the site include: compressed gases for instrument calibration; small amounts of gasoline or diesel fuel; sample containers with hydrochloric acid, nitric acid, or methanol; hexane and the detergent Alconox®. Safety Data Sheets (SDSs) will be kept in a binder on site for each potentially hazardous material (other than waste) that may be brought on site.

PPE: A minimum Level D PPE is (as described in Section 6.1 of the Regional SSHP) is required for all site activities. If indicated by monitoring results, an upgrade to Level C will be made.

Exposure Monitoring: A PID with a 10.6 eV lamp will be used to measure total VOCs. Action levels for upgrades to exposure monitoring and PPE are shown in Table SLOP-3. If after one round of sampling, the PID readings do not indicate the presence of VOCs, monitoring with the PID will be discontinued.

Site Sanitation:

- Drinking water bottled drinking water will be maintained on site for use by all personnel.
- Washing and toilet facilities the nearest facilities will be identified during the morning TSM depending on the location of the day's activities.
- Waste Disposal investigation-derived waste (IDW) generated during the field activities will be classified, handled and disposed in accordance with the Waste Management procedures outlined in the Field Sampling Plan following applicable federal, state, and local regulations. Disposable materials (not classified as hazardous) such as latex gloves, used PPE, aluminum foil, paper towels, and similar items, will be placed and sealed in plastic garbage bags for disposal with sanitary waste from the site.

Applicable Health and Safety Programs and Procedures: Hazard Communication (APP Appendix E), Incident Reporting (APP Appendix F).

Table SLOP-1
Definable Features of Work and Associated Activity Hazard Analyses

Activity	AHA#	Description			
Mobilization/		 Mobilization and demobilization of equipment to the site. 			
De-mobilization/	1	Set-up and take down and staging of equipment.			
General Site Hazards		• General Site Hazards			
Water Level Gauging	2	Measuring the depth to water at monitoring wells.			
F	2	• Collection of groundwater samples from monitoring wells.			
Environmental Sampling 3		• Equipment decontamination.			
		Management of IDW.			
O&M of Monitoring	1	 Inspection of monitoring wells. 			
Wells	7	• Routine maintenance and routine repair.			

Note: AHAs are included in Appendix B of the APP.

Table SLOP-2
Contaminants of Interest and Potential Health Hazard

Contaminant of	Highest Published Exposure Limits for 2012			IP				
Interest (CAS Number)	Observed Concentration	TLV/PEL	STEL/ C	IDLH	(eV)	Health Hazards		
VOCs								
Cis-1,2-dichloroethene (540-59-0)	2320 µg/L (groundwater)	200	-	1000 ppm	9.65	Irritation of eyes and skin; central nervous system depression		
Carbon tetrachloride (56-23-5)	15,100 μg/L (groundwater)	10 Skin A2 Carcinogen	25 ppm	200 ppm Carcinogen	11.47	Irritation of eyes and skin; central nervous system depression; nausea, vomiting; liver, kidney damage; potential carcinogen		
Chloroform	864 μg/L (groundwater)	2		1000	11.42	Irritation, dizziness, nausea		
1,2-Dichroroethane (107-06-2) (ethylene dichloride)	55.6 μg/L (groundwater)	10/50 ppm Skin absorption potential A4 Carcinogen	100 ppm C	50 ppm	11.05	Irritation of eyes and corneal opacity; central nervous system depression; nausea and vomiting; dermatitis; liver, kidney, cardiovascular system damage.		
Tetrachloroethene (127-18-4)	17,100 μg/L (groundwater)	25/100 ppm	100 ppm A3 carcinogen	150 ppm	9.32	Irritation to eyes, nose, throat; flushed face and neck; dizziness and vertigo		
1,1,1,2- Tetrachloroethane (630-20-6)	17.1 μg/L (groundwater)	1ppm		150ppm	11.1	CNS, mucous membranes, eyes and skin		
Trichloroethene (79-01-6)	4210 μg/L (groundwater)	10/100 ppm Skin A2 Carcinogen	25 ppm	150 ppm Carcinogen	9.32	Irritation to eyes and skin; vertigo, headache, fatigue, giddiness, tremors, nausea		
Methylene Chloride (75-09-2)	139 μg/L (groundwater)	50/25 ppm A3 Carcinogen	125 ppm	2,300 ppm Carcinogen	11.32	Irritation of eyes and skin; lassitude; numbness and tingling in limbs; nausea.		

mg/m³ – milligrams per cubic meter

eV - electron volt ppm - parts per million

N/A - not applicable

TLV - threshold limit value (ACGIH) over 8-hr work shift

STEL – short term exposure limit (15 minute)

C- Ceiling limit never to be exceeded

CAS - Chemical Abstract Service registry number

mg/kg - milligrams per kilogram

 μ g/L – micrograms per liter

PEL – permissible exposure limit (OSHA) over 8-hour work shift

IDLH - immediately dangerous to life or health

Skin-can be absorbed directly through the intact skin

Sensitizer—causes chronic sensitization

American Conference of Governmental Industrial Hygienists (ACGIH) Carcinogens

A1—confirmed human carcinogen

A2—suspected human carcinogen, confirmed animal carcinogen hunknown relevance to humans A4—Not classifiable as a human carcinogen

A3—confirmed animal carcinogen with unknown relevance to humans

A4—Not classifiable as a human carcinogen

A5—Not suspected as a human carcinogen

(R) Respirable fraction

Table SLOP-3 Site-Specific Exposure Monitoring and Action Levels

Activity	Location	Frequency	Monitoring Method	Instrument Relative Response	Action Level	Action
Monitoring well sampling	Breathing zone	When well cap is opened	PID with 10.7 eV lamp	50	1ppm	STOP WORK and evaluate options: Ventilate if possible and return to work when air concentrations return to background